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TITLE: THE EVALUATION OF A COMPOSITE TEFLON/ALUMINUM EXPULSION BLADDER FOR USE IN NITROGEN TETROXIDE

MODEL LUNAR ORBITER CONTRACT NO. NAS 1-3800

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ABSTRACT

This report presents the results of an engineering test program designed to demonstrate the capabilities of a composite teflon/aluminum expulsion bladder for use in nitrogen tetroxide. The program encompassed investigation of gas transmission properties, susceptability to repetitive cycling and vibration test, and long-term compatibility with nitrogen tetroxide. Test data verified the suitability of the design in all aspects. A maximum exposure time of 1630 hours (over twice maximum mission time) produced no deterioration of the bladder. The presence of the aluminum interfoil layer reduced the rate of nitrogen gas transmission by approximately two orders of magnitude. Accumulation of six expulsion cycles after subjection to Flight Acceptance and Qualification level vibration test produced no adverse affect on bladder capability. The composite teflon/aluminum expulsion bladder is to be incorporated into the oxidizer tanks of all Lunar Orbiter Flight spacecraft.

KEY WORDS

Nitrogen Tetroxide
Gas Transmission
Vibration
Expulsion Cycles
Teflon/Aluminum Bladder
Nitrogen Saturation
Storage Compatibility
Delamination

REFERENCES

1. D2-100385-1; "Teflon/Aluminum Expulsion Bladder Development Test Program", J. Carhart, dated 10 November, 1965

2. D2-100623-1; "An Investigation of Titanium-Alloy Propellant Tank Behavior under Conditions of Long-Term Operation", J. Carhart, dated 10 February, 1966

3. Lunar Orbiter Program Directive 32, R1; "OPERATIONS - Spacecraft Oxidizer Tanks", R. J. Helberg, dated 20 January, 1966

4. D2-100638-1; "Oxidizer Bladder Permeability - Lunar Orbiter Spacecraft", A. E. Gemear, to be released.

5. 10-70056; "Procurement Specification, Tank, Oxidizer", Revision F, dated 1 February, 1966

6. Marquardt Report 6180; "Final Report on Supplemental Qualification Program for the Lunar Orbiter Velocity Control Engine", dated 17 January, 1966

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1.0 INTRODUCTION

Operation of the Lunar Orbiter Velocity Control Subsystem rocket engine has been shown to be of marginal stability when nitrogen-saturated oxidizer is employed. Unstable combustion at a frequency of 340-360 cps has been observed during engine-vendor tests, and during the Subsystem Design Verification/Reliability Demonstration test program. Test data has shown that under conditions of oscillatory operation, engine performance is degraded on the order of 10-15%, and that the thrust oscillations may induce structural vibration characteristics that may have an adverse affect upon sensitive flight control equipment.

Study efforts revealed that the more economical approach to the problem solution, and one that would have the least impact on spacecraft scheduling, would be to significantly reduce the rate at which the nitrogen pressurant diffused through the expulsion bladder and dissolved into the oxidizer. In the Lunar Orbiter mission, the last velocity change maneuver occurs approximately 18 days after launch, and the Velocity Control Subsystem is no longer operative beyond that point.

Further investigation revealed that the Jet Propulsion Laboratory had sponsored preliminary development effort of a composite teflon/aluminum expulsion bladder for the Surveyor spacecraft. The limited amount of test data available indicated that the aluminum foil interlayer provided a substantial barrier to nitrogen gas transmission, and that the oxidizer saturation level after 18 days of exposure (estimated) was quite low.

The test data were encouraging to the point that procurement of several test bladders was authorized. Test bladders were purchased from the Dilectrix Company which also supplies the standard all-teflon expulsion bladders for Lunar Orbiter, Apollo, LEM, Gemini, etc. This report presents the results of a comprehensive Boeing Company test program to evaluate a composite teflon/aluminum expulsion bladder for use in the Lunar Orbiter VCS oxidizer propellant tanks. The scope of the program encompassed, and emphasized, the following bladder characteristics and capabilities: 1) compatability with nitrogen tetroxide under long-term storage, 2) repetitive expulsion cycles, 3) gas transmission characteristics, and 4) vibration test compatability.

2.0 SUMMARY

A composite teflon/aluminum expulsion bladder has been demonstrated as suitable for use in nitrogen tetroxide. The major goal of the program has been to demonstrate that the presence of the aluminum interfoil will sufficiently impede the transmission of nitrogen to minimize oxidizer saturation at the conclusion of Velocity Control Subsystem operation. The design shall also be capable of withstanding repetitive cycling, thermal environment, and launch-induced vibration spectrums. The results of the test program show that the bladder design fulfills all objectives and requirements.

Gas transmission properties of the teflon/aluminum bladder were evaluated by periodically sampling the oxidizer to determine the amount of nitrogen in solution. A total of 26 permeation measurements were conducted on three seperate bladder assemblies. The resultant data indicate that the nitrogen saturation level at the conclusion of a 32 day mission profile will be nominally 19%. In the same time period, the saturation level with a 6-mil, all-teflon bladder is approximately 100% Data indicate a nitrogen transmission rate of 0.006-0.01 scc/hr/in^2 for a teflon/aluminum bladder, and a transmission rate on the order of 0.8 scc/hr/in^2 for the standard bladder.

Expulsion capability has been demonstrated by subjecting four test units to a total of 13 expulsion cycles, nine of which were to the 98% level. One unit accumulated a total of six expulsion cycles. Expulsion tests were conducted at temperature extremes of 36.5°F and 86.9°F. No degradation or damage was observed as a result of repeated bladder flexing.

Four test units have been exposed to nitrogen tetroxide for an accumulated interval of 3658 hours. Maximum exposure time on one unit was 1630 hours, a value that is more than twice maximum VCS mission operating time. No bladder deterioration was observed in that time period, though a few minute areas of localized delamination were noted. The delamination produced no detectable variation in gas transmission or general leakage characteristics. Storage tests were conducted at 50-85°F.

Four test units were subjected to launch-induced vibration spectrums; all were tested to Flight Acceptance levels, and two were tested at the Qualification level. One test unit exhibited excessive leakage at the conclusion of Qual-level vibration test. An inspection of the unit revealed a permanent twist with respect to the propellant standpipe, and it is believed that this failure may be attributed to slightly improper installation aggravated by vibration test. All other test units exhibited zero leakage at the conclusion of their vibration test.

There is definitive evidence to indicate that the test bladders supplied directly to The Boeing Company for purposes of this test program have been undersized. The Dilectrix Company originally estimated that post-curing shrinkage of the composite bladder would not be as great as that of an all-teflon unit; the fabrication mandrel was shortened 1% to accommodate this difference. Further data indicates that the shrinkage values are nearly equal. Inspection upon receipt revealed a fine network of light patterns, indicative of minute aluminum foil cracking, in almost all test units. Corrective control procedures have been instituted to prevent recurrence. Six test bladders and one production unit were sectioned and measured to evaluate material thickness; the average thickness of the test units was 5.98 mils, and the average thickness of the production bladder was 5.82 mils.

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2.0 PROGRAM SCOPE

As indicated in the Reference 1 program plan, the teflon/aluminum expulsion bladder development effort consisted of several investigative areas. Test programming emphasized determination of nitrogen gas transmission rate, expulsion cycle capability, compatability with nitrogen tetroxide over a long-term period at operating conditions, and the susceptability of the bladder to the Lunar Orbiter vibration environments. Several test sites were involved in the program: Vibration tests were conducted at the Kent Space Center; storage and expulsion tests with actual propellant were conducted at the Tulalip Test Site; gas transmission characteristics were evaluated in the Materials & Processes Laboratory at Kent.

A brief summation of the test program is presented in Table I. The material reported herein is for complete tank/bladder assemblies. A complimentary test program has been conducted by the M&P Research organization and is reported separately in Reference 4: that program consisted of gas transmission, compatability, and peel strength tests on small samples of bladder material; i.e., coupons. Although basically an engineering demonstration test program, the teflon/aluminum bladder tests have been oriented to a level comparable to a qualification-type program.

TABLE I

TEST MATRIX

TEFLON/ALUMINUM BLADDER DEVELOPMENT

Unit	Test Program
Oxidizer Tank S/N 9 Bladder S/N 121-3M	1. Conduct 8-day storage test at 240 psig and 80°F. 2. Determine N_2 content on 4th and 8th day. 3. Conduct one complete expulsion cycle, 98%.
Oxidizer Tank S/N 4 Bladder S/N 123-3M	1. Subject to FAT vibration with simulated propellant. 2. Expel 80 lbs of oxidizer at temperatures of 40°F and 85°F. 3. Conduct 98% expulsion cycles at 40°F and 85°F.
Oxidizer Tank S/N 10 Bladder S/N 149-3M	1. Conduct FAT and Qual-level vibration tests with simulated oxidizer. 2. Expel 80 lbs of oxidizer at 40°F and 85°F. 3. Conduct 98% expulsion cycles at 40°F and 85°F. 4. Conduct two mission simulation tests (32 days each) in real time. Perindically determine N_2 content.
Oxidizer Tank S/N 4 Bladder S/N 152-3M	1. Subject to FAT vibrating. 2. Conduct two mission simulation tests in real time (32 days each). Periodically determine N_2 content.

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4.0 TEST FACILITIES

A brief description of test support facilities utilized in the program are presented in this section. General operational techniques and instrumentation system characteristics are included. Storage and expulsion tests with nitrogen tetroxide, because of its high toxicity, were conducted at the Tulalip Test Site. Vibration tests were conducted at the Kent Space Center.

4.1 STORAGE & EXPULSION TEST - TULALIP

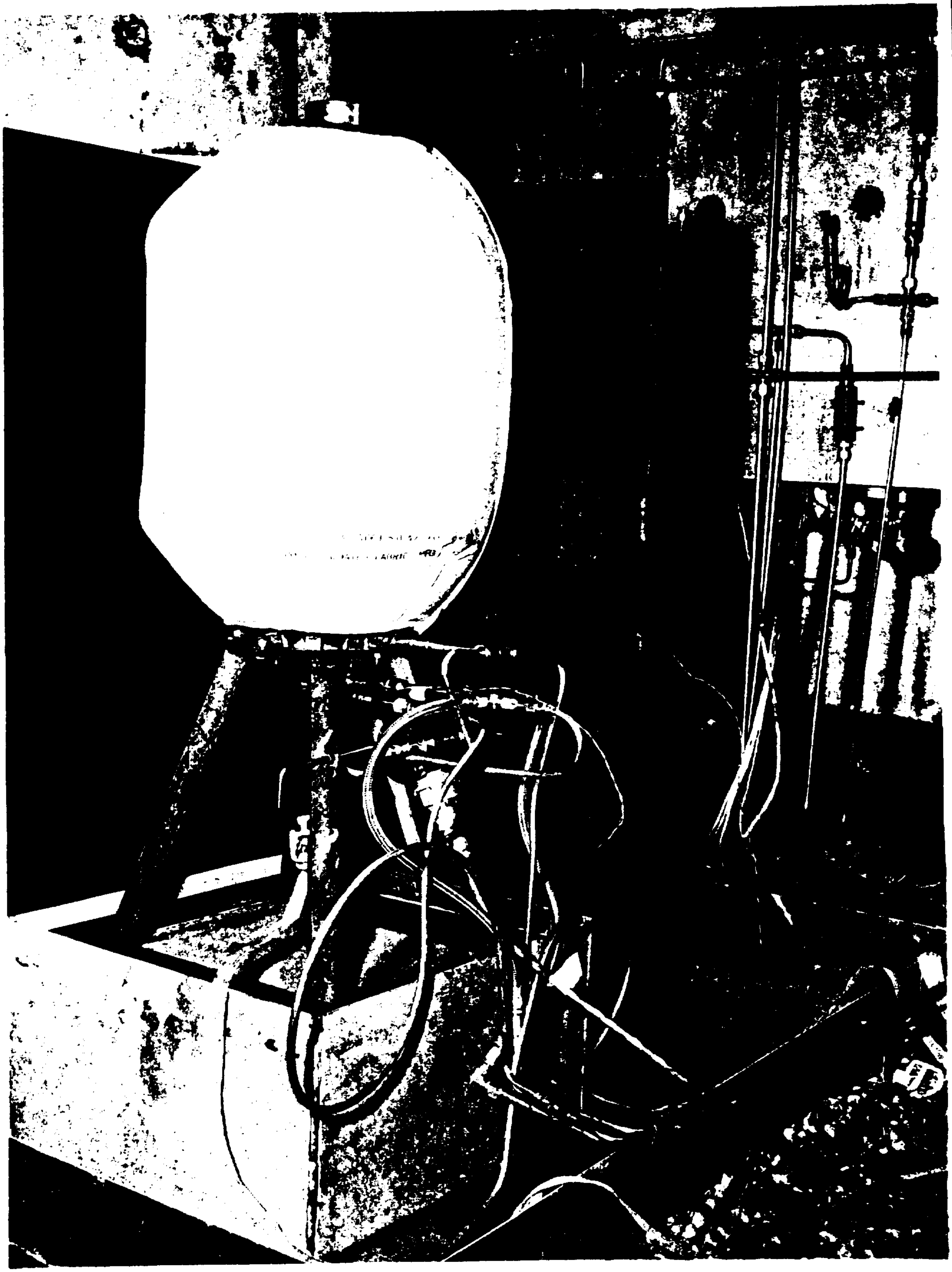
Those phases of the program requiring long-term exposure and/or the expulsion of nitrogen tetroxide were conducted in Area 5 at the Tulalip Test Site. The equipment and techniques employed were similar or identical to those used in the titanium-alloy propellant tank investigations (Reference 2).

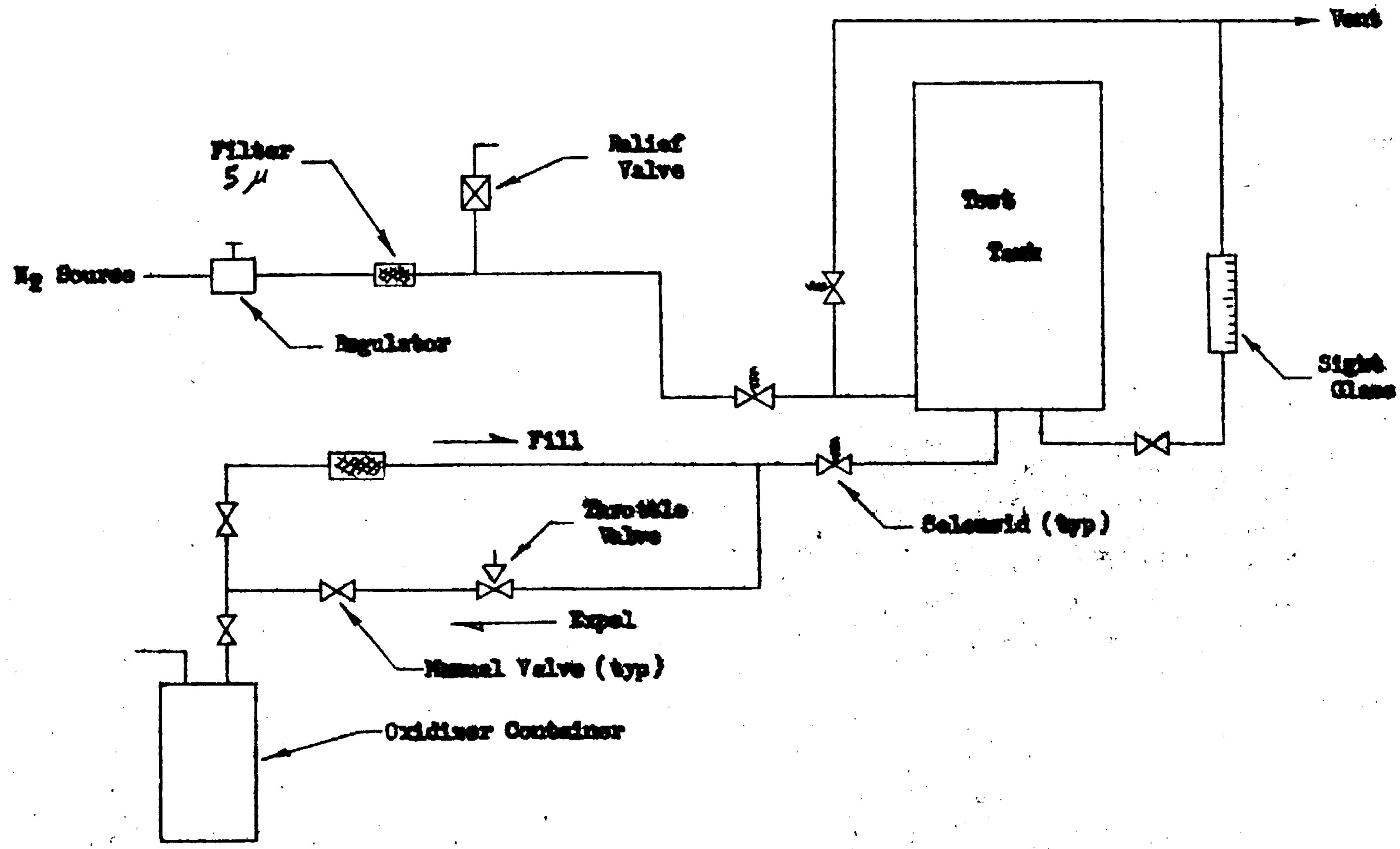
4.1.1 Test Installation

The tanks were mounted on a facility-supplied stand with the longitudinal axis vertical and the tank outlet down. Figure 1 shows a typical installation; the protective shipping cover was removed prior to test. An insulated cover was positioned over the tank to provide the necessary thermal control. The desired temperature environment was maintained by a thermostatically controlled, electrically operated hot-air blower (75,000 BTU Coates Air Heater). The thermostat senses and controls the heater's outlet temperature, not the temperature within the test cell; hence, the environmental temperature will tend to fluctuate somewhat depending on the effectiveness of the insulation and the extremes of ambient temperature. As will be shown in Section 6.3, the environmental temperature seldom varied more than 5°F in a 24-hour period.

Propellant loading operations were conducted by transferring directly from the commercial shipping containers in conjunction with facility-supplied lines and valving. Lunar Orbiter ground servicing equipment was not available for this test program. The propellant tank was filled with nitrogen tetroxide (at a transfer pressure of approximately 25 psig) until overflow was observed in the liquid vent line sight glass. The liquid vent valve was then closed, the tank pressurized to 25 psig, and three pounds of oxidizer were off-loaded back into the shipping cylinder to provide the necessary ullage volume. Propellant weights were determined by means of a facility-supplied platform scale readable to about 0.5 pounds. No difficulties were encountered in these operations, and each tank was loaded in 15-20 minutes.

Each tank was equipped with three solenoid-operated valves for pressurization, venting, and propellant loading/expulsion. A manual throttle valve was included to achieve the desired flow control. A simplified schematic of the test configuration is shown in Figure 2. The pressurization and propellant valves were of the normally-closed type, and the vent valve was of the normally-open type; thus, in the event of an electrical failure, the pressurization and propellant valves would automatically close (if they were open) and the vent valve would open. The normal operating mode after oxidizer loading was completed (for storage tests) was to pressurize the tank and then close the pressurization valve; repressurization would occur as required to account for small leaks in the facility system and/or pressurization gas dissolving into the propellant. During expulsion test, the pressurization valve remained open.





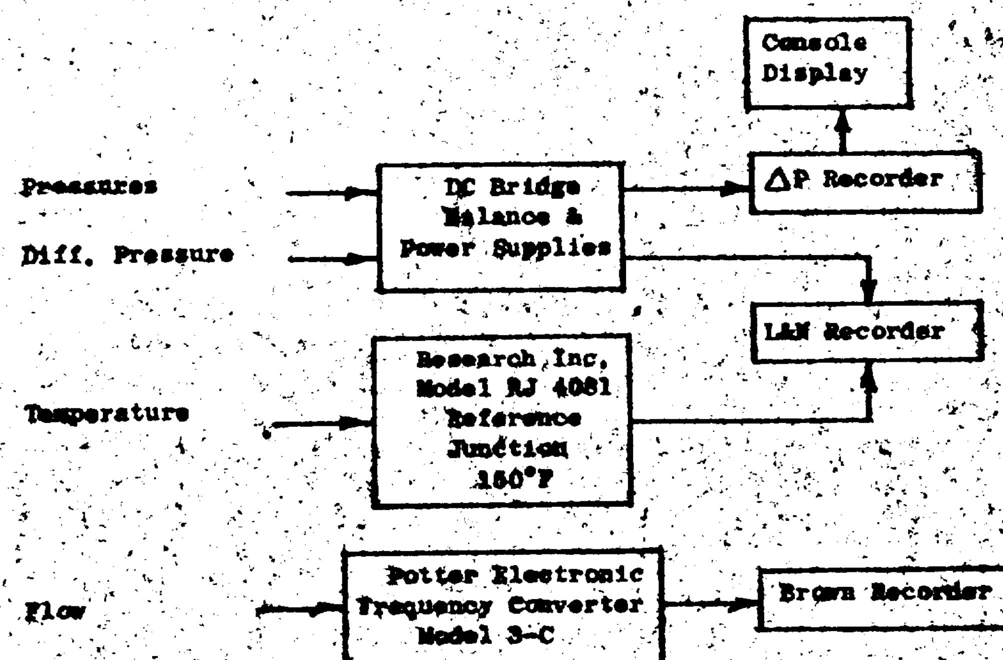
STORAGE/EXPULSION TEST SCHEMATIC

Figure 2

4.1.2 Instrumentation

During storage phases of the program, tank pressure and temperature were recorded on a continuous 24-hour basis. The parameters were recorded on a Leeds & Northrup, Model G, recorder. This unit is a multi-point recorder in that several data channels may be recorded on the same instrument. This is accomplished by means of a stepping switch and index wheel that stamps a "point" and channel number for each data parameter. The L&N recorder has 16-channel capability and the time increment between successive channels is approximately 30 seconds. During storage test the recorder was controlled by a camoperated timer; every 40 minutes, the recorder was turned on for about five minutes. During expulsion tests, the timer was bypassed and the recorder operated for the duration of the test. The pressure loss across the tank was recorded on a standard Leeds & Northrup strip chart recorder with a display on the control console; this data system was operative only during an expulsion cycle. Oxidizer flowrate during expulsion was to be recorded on a Honeywell "Brown" recorder as a d-c trace.

A simplified block diagram of the instrumentation systems is shown in Figure 8. The accuracy of the pressure and differential pressure measurements are estimated to be on the order of 0.5-1.0%, temperature measurement accuracy is 1-2%, and the flow measurement accuracy was 3-5%. Instrumentation discrepancies observed during the program were failure of the flow measurement, and an 18-hour malfunction of the L&N multi-point recorder. A special turbine-type flowmeter was purchased from the Potter Aeronautical Corporation (Model No. 3/8"-5790;



INSTRUMENTATION SYSTEMS
BLOCK DIAGRAM

Figure 8

Serial No. BOE 3/8"-26) with the specific requirement that the instrument be compatible for long-term usage in nitrogen tetroxide. No recording was obtained on the initial expulsion cycle after five days exposure to the oxidizer. Removal and inspection of the meter revealed that the turbine was apparently "frozen" in its bearings. The flowmeter was returned to the vendor. On subsequent tests, average flow was determined from the expelled weight and elapsed time. An amplifier failed in the L&N recorder on 1 March and approximately 18 hours of pressure/temperature data were lost.

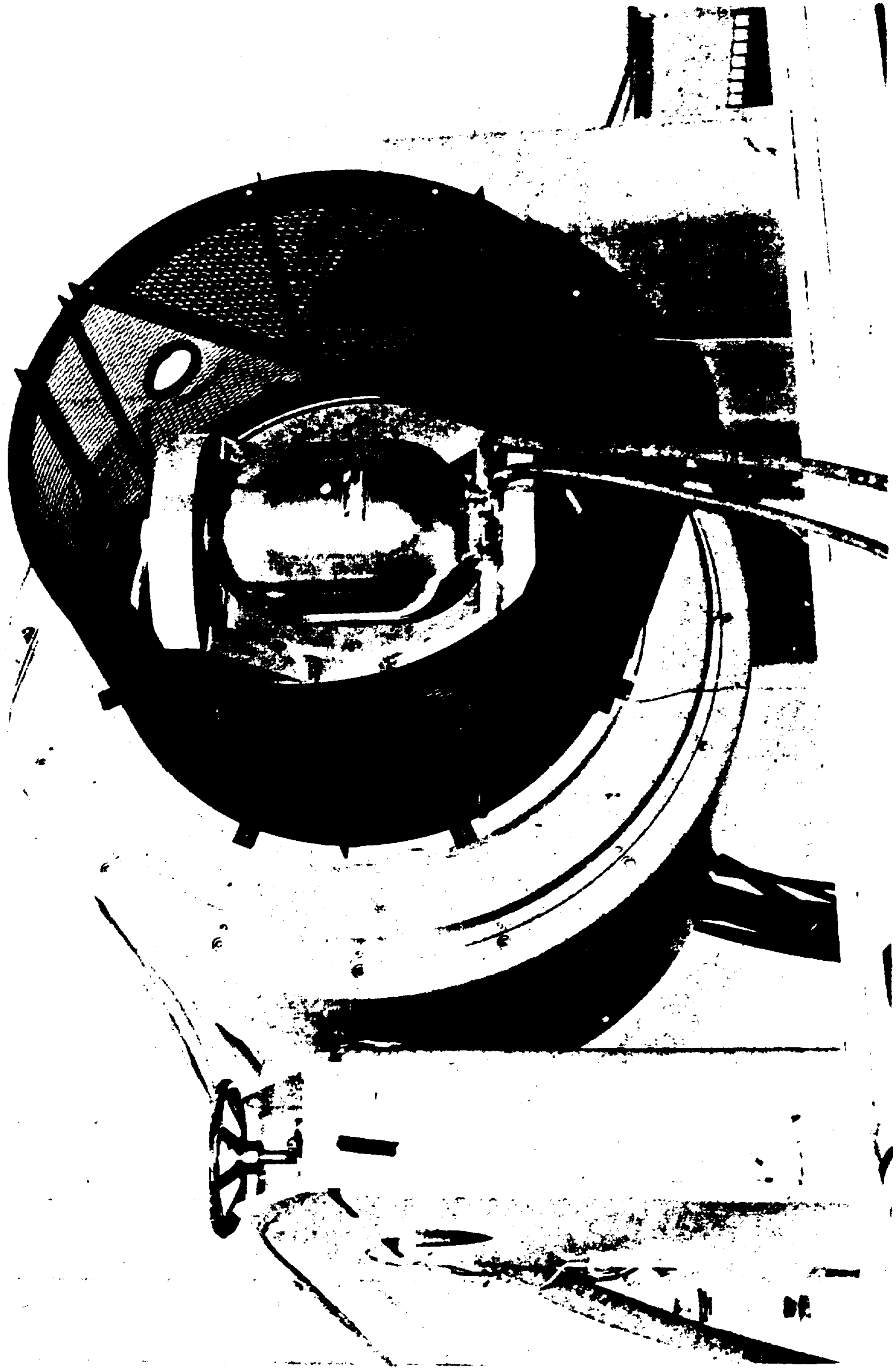
4.2 VIBRATION TEST - KENT

Vibration test of the various tank assemblies were conducted at the Kent Space Center, Building 18-24. The test fixtures were fabricated from designs obtained from Bell Aerosystems to insure comparable response characteristics. The test equipment consisted of a Ling 249 vibration exciter driven by a Ling 175 KVA amplifier in conjunction with several units of peripheral support equipment; i.e., tape recorder, sweep oscillators, ARDE 45 automatic equalizer, etc. Figure 4 presents a photographic view showing tank installation on the vibration exciter for Z-axis testing. Figure 5 shows a similar view for X-axis test prior to installation of the protective cage. The hoses leading to the test tank are for liquid transfer and pressurization.

Prior to initiating a vibration test, the tank is filled with a mixture of Freon and methanol to simulate the nitrogen tetroxide. After overflow is achieved, 1300 cubic centimeters of liquid are off-loaded to provide the ullage volume; the tank is then pressurized to 40 psig. After completing a test, the simulation liquid is drained from the tank by pressurizing through the liquid vent line; this defueling technique avoids cycling the expulsion bladder.

During test, acceleration data are recorded on magnetic tape (Ampex FR 1300 tape recorder); input vibration was monitored by an Endevco 2221D accelerometer, and the response of the tank assembly was monitored by an Endevco 2226 accelerometer. The data is recovered by replaying the tape through suitable electronic instrumentation equipment. Sinusoidal spectrum test data are scanned and the maximum acceleration levels automatically plotted as a function of the frequency. Random spectrum data are scanned by a power spectral density analyzer which plots the data in terms of the square of the acceleration level (to avoid negative numbers) as a function of frequency; the power spectral density of a non-periodic acceleration function is the average acceleration in a one cps frequency band plotted as a continuous function of frequency.

Figure 4



VIBRATION TEST, Z-AXIS

Figure 5

VIBRATION TEST, X-AXIS

5.0 BLADDER TEST HISTORY

Nine teflon/aluminum expulsion bladders were procured for engineering test purposes. The composite bladder is essentially identical to the standard all-teflon units with the addition of a 0.25-mil layer of 1100-series aluminum foil to provide the gas transmission barrier. From the liquid side out, the bladder is constructed in the following manner: 2 mils of TFE teflon; 1 mil of FEP teflon; 0.25 mils of aluminum foil; 3 mils of FEP teflon. All layers are bonded together. Table II briefly summarizes the test history of each unit.

TABLE II

BLADDER TEST HISTORY

Bladder S/N	Tank S/N	History
-	-	Bladder shipped in unpressurized aircraft - found to be ruptured upon arrival. Return to vendor.
121-3M	9	Subjected to 208 hours storage in nitrogen tetroxide at 240 psi and 80°F. One complete expulsion.
123-3M	4	Subjected to FAT vibration. Subjected to two 90% and two 98% expulsion cycles.
122-3M	-	Defective upon arrival; return to vendor.
124-3M	10	Subjected to FAT and Qual vibration. Excessive leakage noted.
149-3M	10	Subjected to FAT and Qual vibration. Subjected to four expulsion cycles, followed by two real-time mission simulation tests.
150-3M	-	Small puncture noted prior to tank installation; time and origin of puncture unknown.
151-3M	-	Failed due to overpressurization in leak test; human error.
152-3M	4	Subjected to FAT vibration. Subjected to two real-time mission simulation tests; two expulsion cycles.

Being engineering development units, the test bladders were purchased without benefit of rigorous quality control inspection and were installed in the tanks by Boeing personnel. The above listing emphasizes the necessity for extreme caution in handling, shipping, and testing an expulsion bladder; four bladders were either damaged upon receipt or prior to installation into a tank shell - a mortality rate of 45%.

A small degree of shrinkage is encountered as the bladder is cured. The Dilectrix Company had estimated that the shrinkage of the composite bladder would not be as great as for an all-teflon bladder, and the fabrication mandrel was shortened 1% to compensate for the estimated difference. More recent data indicates that the shrinkage of the teflon/aluminum bladder is more nearly equal to that of the all-teflon bladder, and there is evidence to suspect that test bladders were undersize. Inspection upon receipt revealed a fine network of light patterns, indicative of minute aluminum foil cracking, in almost all test units. Subsequent tank installation and testing served to aggravate the condition. Boeing and Bell Aerosystems have instituted the necessary inspection and control procedures to prevent recurrence of the problem.

Several test bladders and one production unit were examined to ascertain material thickness; the minimum thickness of the teflon/aluminum bladder should be 6.25 mils. Four longitudinal "gores" were removed from each unit, and nine measurements were made on each gore. Thickness measurements were made with a Pratt & Whitney "Super Micrometer", Model G-2100, at an anvil force of one pound. The following summarizes the results:

__Average Bladder Thickness__

S/N 121-3M.	6.21 mils
S/N 123-3M.	6.26 mils
S/N 149-3M.	5.94 mils
S/N 151-3M.	5.94 mils
S/N 152-3M.	5.77 mils
S/N 154-3M.	5.82 mils
S/N 153-1	6.52 mils

Bladder S/N 153-1 was the production unit.

6.0 TEST PROGRAM RESULTS

The engineering test program devised to evaluate the composite teflon/aluminum expulsion bladder for use in the Lunar Orbiter oxidizer tanks encompassed several areas of investigation. The areas of test included 1) expulsion cycling, 2) gas transmission rate determination, 3) long-term exposure and compatability, and 4) vibration test environment. Most tank/bladder assemblies were subjected to all of the above test conditions. The test program results presented in the subsequent sections are arranged in terms of the primary investigative area rather than a chronological summary on the basis of tank assembly identification.

6.1 EXPULSION CHARACTERISTICS

In the operational mission, the VCS propellant tank bladders are subjected to only one complete expulsion cycle. A determination of teflon/aluminum bladder cyclic capability has been accomplished by subjecting two units to four expulsion cycles each. Additional expulsion data has been accrued in the course of storage and mission simulation testing, and will be summarized as a portion of that investigative area (Section 6.3). The "expulsion cycle" phase of the test program subjected the test unit to two 90% expulsion cycles and two 98% cycles; the tests were conducted at high and low temperature extremes of 85°F and 40°F. A 90% cycle consisted of expelling 80 pounds of oxidizer; a 98% cycle, by definition, is conducted until the pressure loss across the tank assembly exceeds 2 psi.

6.1.1 Tank S/N 4; Bladder S/N 123-3M

After completing FAT-level vibration testing at the Kent Space Center, Tank S/N 4 was delivered to the Tulalip Test Site on 2 December for the programmed series of expulsion tests. The required four cycles were accomplished without incident. Table III summarizes the test conditions as actually recorded.

Table III

BLADDER S/N 123-3M
EXPULSION TEST SUMMARY

Cycle Number	Pressure, psig	Temperature, °F	Flowrate, lbs/sec	Weight, lbs.	Date
1	190.6	39.4	–	80	12-2
2	184.3	85.6	0.103	80	12-2
3	182.2	37.9	0.0825	91	12-6
4	186.8	85.1	0.099	91	12-6

The above values of pressure, temperature, and flowrate are average for the test. Following the second and fourth expulsion cycles, a bladder leak test was conducted at an internal pressure of 10 psig. The measured leak rates were zero and 3 scc/15 minutes, respectively. The tank assembly was flushed with a Freon/methanol solvent on 7 December and returned to the KPC for disassembly and inspection. Oxidizer was present in the tank assembly for a total of 118 hours.

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After removal of Bladder S/N 128-3M from the tank, it was noted that there were several hair-line cracks in the outer teflon FEP lamina and the aluminum foil. Figure 6 presents an overall photographic view of the bladder and tank; Figures 7 and 8 are close-up views of the major hair-line cracks. Viewing the bladder internally with strong back-lighting reveals a network of minor cracking not normally visible from an external inspection; this is shown in Figure 9. As discussed in Section 5.0, the observed cracking cannot be positively attributed to the repetitive cycling - the bladder may have been slightly undersized. The measured leak rate being within specification indicated that the underlying FEP-TFE lamina was still intact. No other discrepancies were observed.

6.1.2 Tank S/N 10; Bladder S/N 149-3M

After successfully completing FAT and Qual-level vibration testing, Tank S/N 10 was delivered to Tulalip on 18 December. The unit was subjected to the programmed expulsion cycle testing without difficulty. A summarization of test data are presented in Table IV.

Table IV

BLADDER S/N 149-3M
EXPULSION TEST SUMMARY

Cycle Number	Pressure, psig	Temperature, °F	Flowrate, lbs/sec	Weight, lbs	Date
1	188.7	36.5	0.09	80	12-18
2	191.3	86.5	0.1295	80	12-19
3	193.5	44.0	0.11	90	12-19
4	201.0	86.8	0.123	90.5	12-20

A plot of a typical low temperature 98% expulsion cycle is presented in Figure 10. This plot shows temperature, pressure, pressure loss across the tank assembly, and the quantity of propellant remaining, all as a function of test time. The measured pressure loss is negative for the majority of the test as the transducer is below the tank and is reflecting the propellant head pressure (see Figure 1). A similar presentation for a high temperature expulsion is shown in Figure 11. The pressure loss characteristics in the final stages of expulsion are shown on an expanded time scale in Figure 12; these characteristics are comparable to Bell Aerosystems data for the standard all-teflon expulsion bladder. As the flowmeter was inoperative, the propellant quantity data shown in Figures 10-12 are calculated values based on the assumption that the average flowrate (Table IV) was constant throughout the test.

Bladder S/N 149-3M was leak tested following the second and fourth expulsion cycles; zero leakage was measured on both occasions. Tank S/N 10 was flushed and returned to the MPC on 20 December. Oxidizer had been present in the assembly for 45 hours. Upon disassembly, Bladder S/N 149-3M was found to be in excellent condition; only minute areas of incipient aluminum deterioration were noted in locations of severe folding and creasing. The condition of this unit tends to support the hypothesis that the discrepancies observed in Bladder S/N 128-3M were the result of that unit being undersized. Bladder S/N 149-3M was re-installed in Tank S/N 10 and returned to Tulalip for mission simulation

Figure 6

BLADDER S/N 123-3M DISASSEMBLY

Figure 7

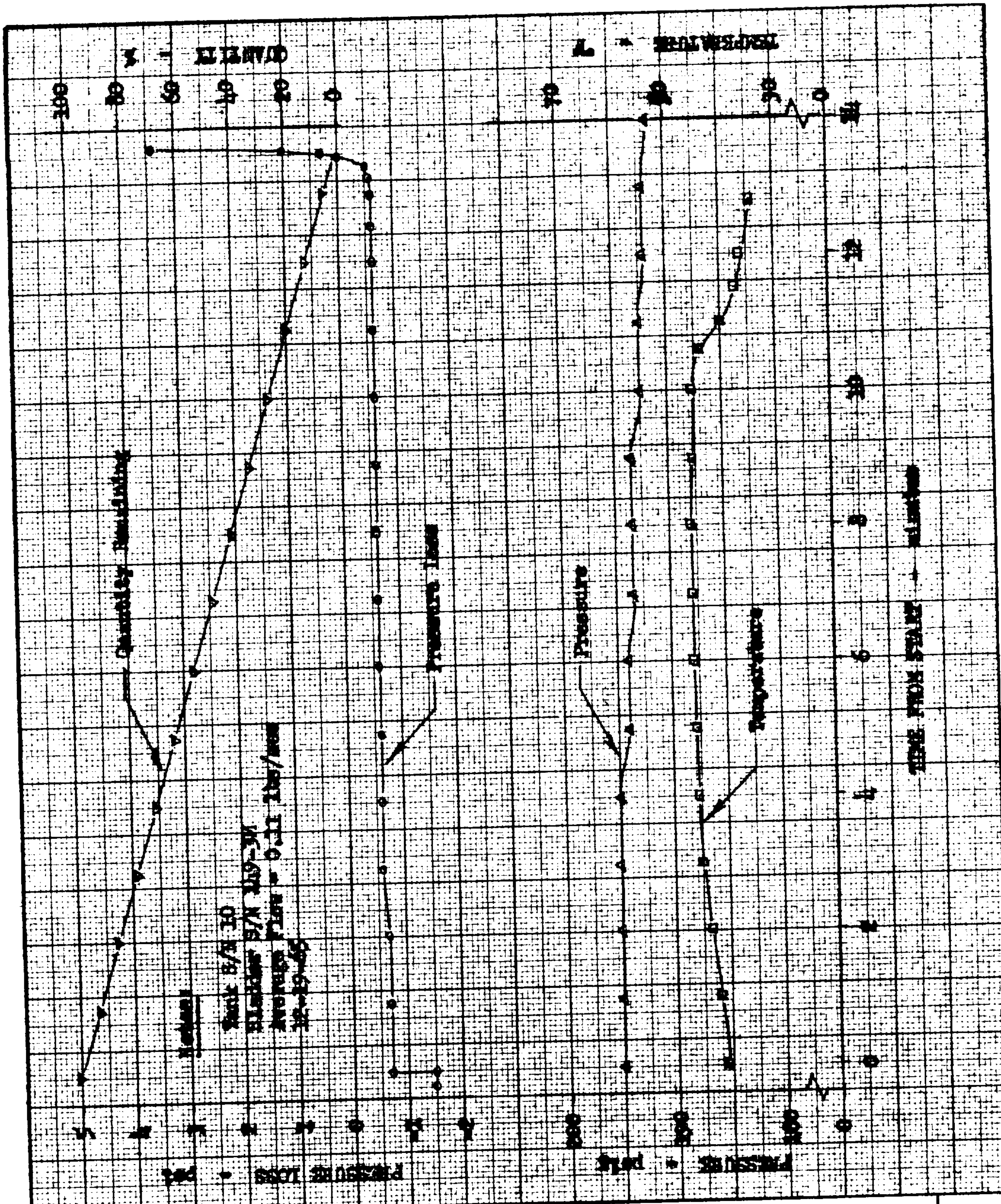
BLADDER S/N 123-3M CRACKING

Figure 8

TEFLON & ALUMINUM SEPARATION

Figure 9

FORWARD EXTREMITY CRACKING
BACKLIGHTED EXTERNALLY



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CALC	JC	1-5-66			TEFLON/ALUMINUM EXPULSION BLADDER EXPULSION CYCLE CHARACTERISTICS LOW TEMPERATURE	Fig. 10
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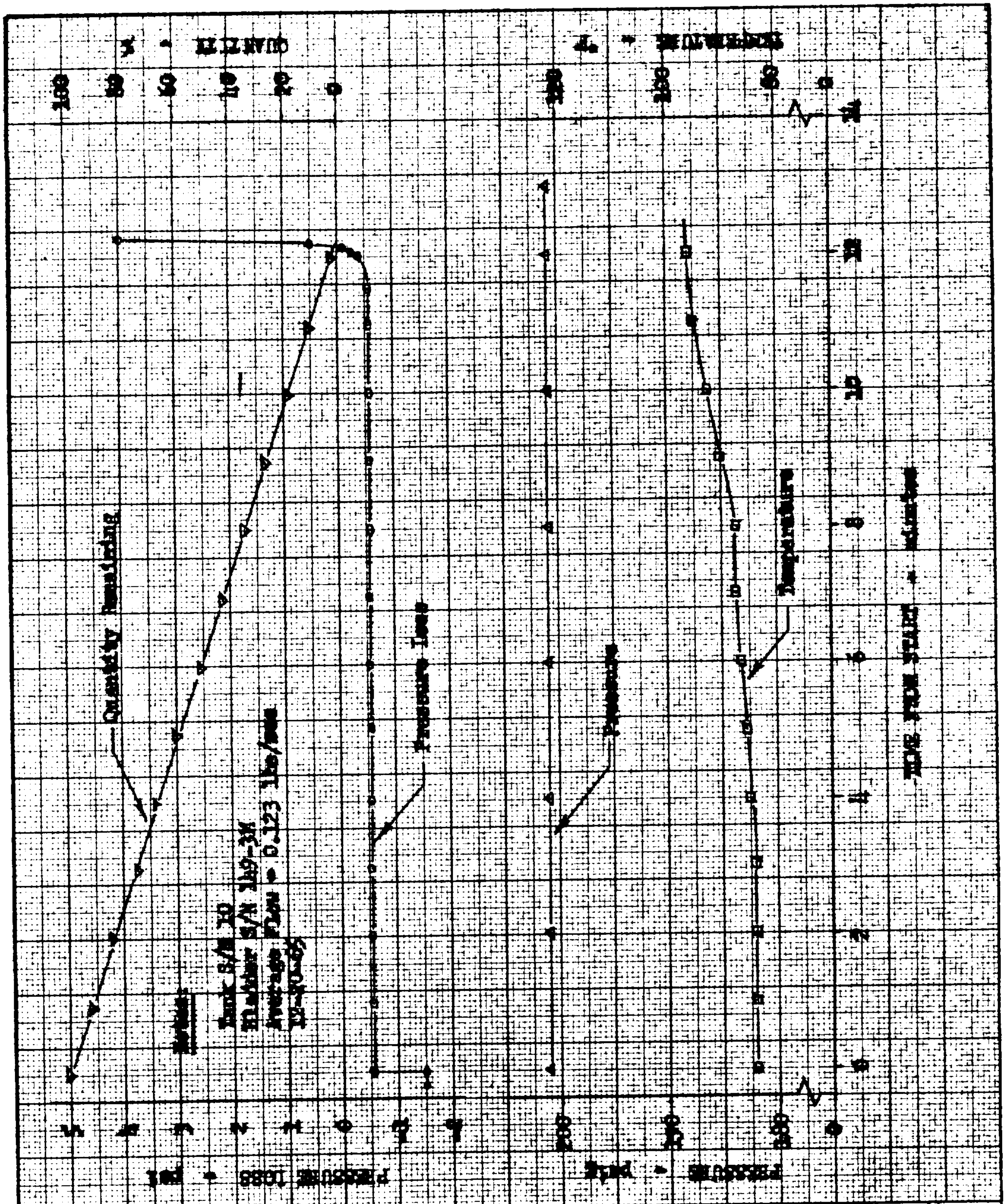

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CALC	JC	1-4-66			TEFLON/ALUMINUM EXPULSION BLADDER EXPULSION CYCLE CHARACTERISTICS HIGH TEMPERATURE	Fig. 11
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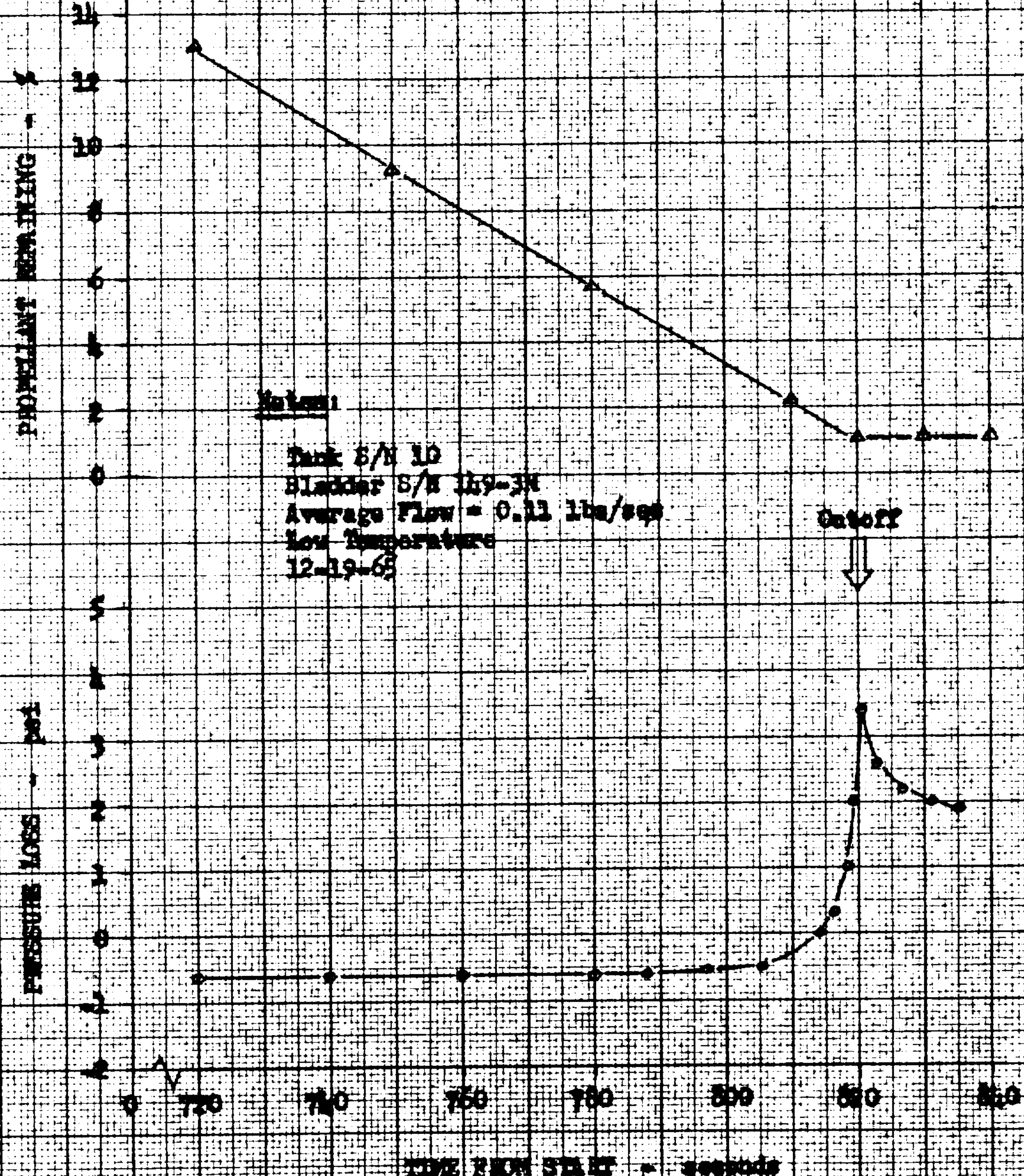

	INITIALS	DATE	REV BY INITIALS	DATE	TITLE	MODEL
CALC	JC	1-5-66			PRESSURE LOSS PROFILE AT HIGH EXPULSION EFFICIENCY	Fig. 12
CHECK						
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testing (see Section 6.3).

6.2 PERMEATION & GAS TRANSMISSION DATA

The nitrogen gas transmission properties of the composite teflon/aluminum expulsion bladder were evaluated on two levels: 1) complete bladder assemblies as installed in the tank, and 2) on small samples or "coupons". Transmission data on all-teflon coupons were also obtained for verification of all-teflon predicted data, and to provide a comparison for teflon/aluminum data. Data from these test efforts are reported accordingly.

6.2.1 Experimental Technique

The gas transmission properties of the expulsion bladder material were measured by determining the quantity of nitrogen in solution with nitrogen tetroxide as a function of time. The same measurement technique was employed for both the complete bladder tests and the coupon tests. The method is reported in detail in Reference 4 and is briefly summarized herein.

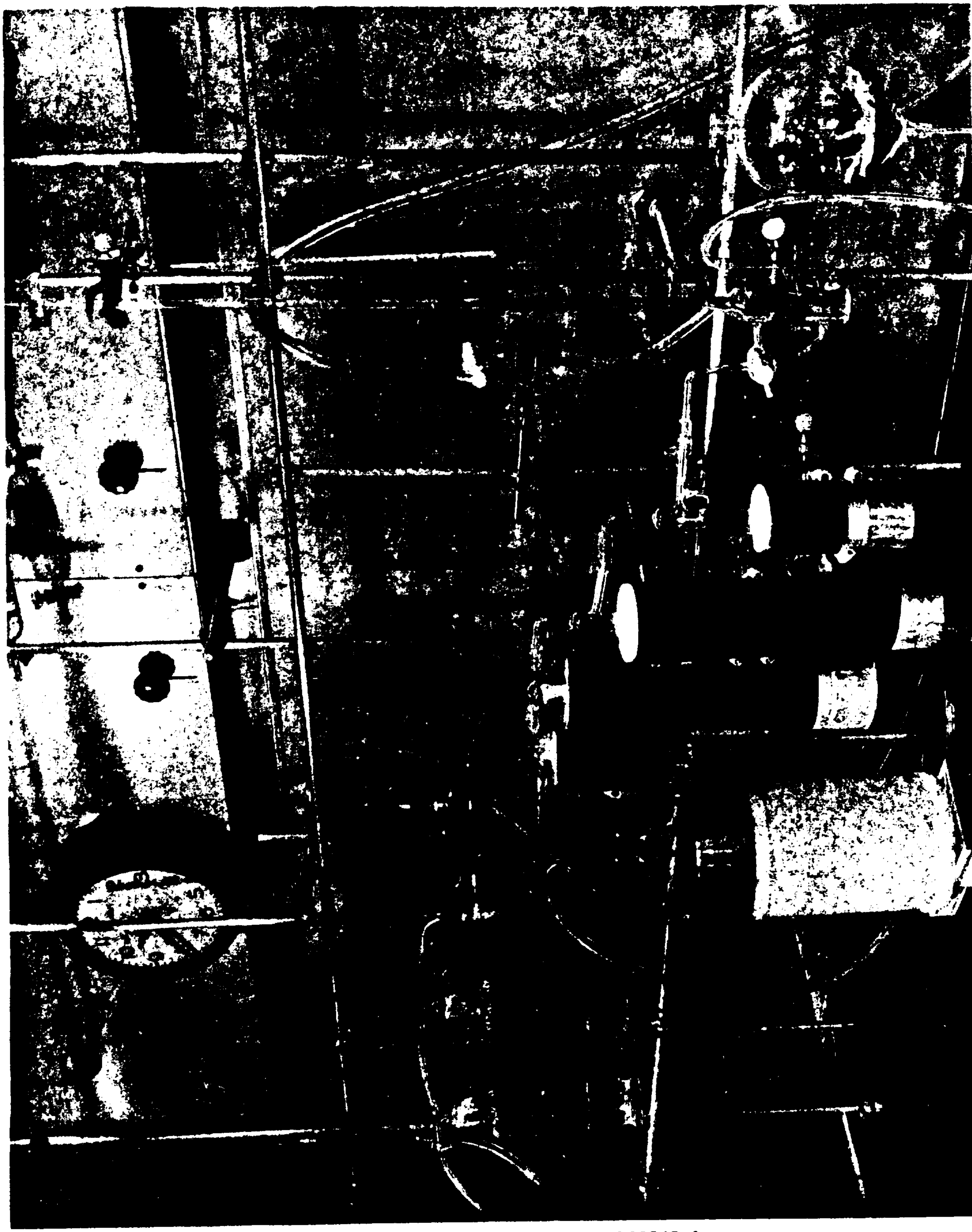
Gas content determination tests were conducted in the Materials and Processes Laboratory, Organization 2-5543, at the Kent Space Center. The chemical apparatus test set-up is shown in Figure 13. The nitrogen-saturated oxidizer is slowly expelled from its container by carbon dioxide and is passed through a 4-stage "cold trap". The cold trap serves to separate out the nitrogen tetroxide. After leaving the final cold trap stage, the gas mixture is bubbled through a burette containing a sodium hydroxide solution which absorbs the carbon dioxide. The volume of the nitrogen that was in solution with the nitrogen tetroxide, plus trace contaminants (oxygen, air, nitric oxide, etc.), are measured in the burette. The contents of the burette are then evacuated into a storage vessel. The vessel is then connected to a gas chromatograph which records the relative amounts of the gas mixture constituents. The relative volumes indicated by the chromatograph and the total volume measured in the burette yield the absolute amount of nitrogen which is then corrected to standard pressure and temperature (760 mm of mercury; 0°C).

6.2.2 Complete Bladder Results

The rate at which a gas diffuses through a thin membrane is a function of several factors; 1) the molecular weight of the gas, 2) the partial pressure gradient across the membrane, and 3) the molecular structure of the membrane. The physical properties of nitrogen tetroxide are such that a relatively large amount of nitrogen can go into solution; hence, initially there will be a large partial pressure gradient. The molecular structure of a plastic such as teflon is relatively permeable to the passage of gaseous nitrogen. The end result of these characteristics is that at Lunar Orbiter operating conditions, the level of nitrogen saturation in the oxidizer exceeds the 90% value after 8-9 days of exposure. During VCS engine operation, the propellant pressures at the engine are less than in the propellant tanks; hence, the nitrogen will tend to come out of solution and nucleate into bubbles of varying size. Test data indicate that the bubble formation associated with saturation concentrations in excess of 60-70% (Reference 5) will cause the MA-100 engine to become unstable.

USE FOR TYPEWRITTEN MATERIAL ONLY

Figure 13



NITROGEN CONTENT ANALYSIS EQUIPMENT

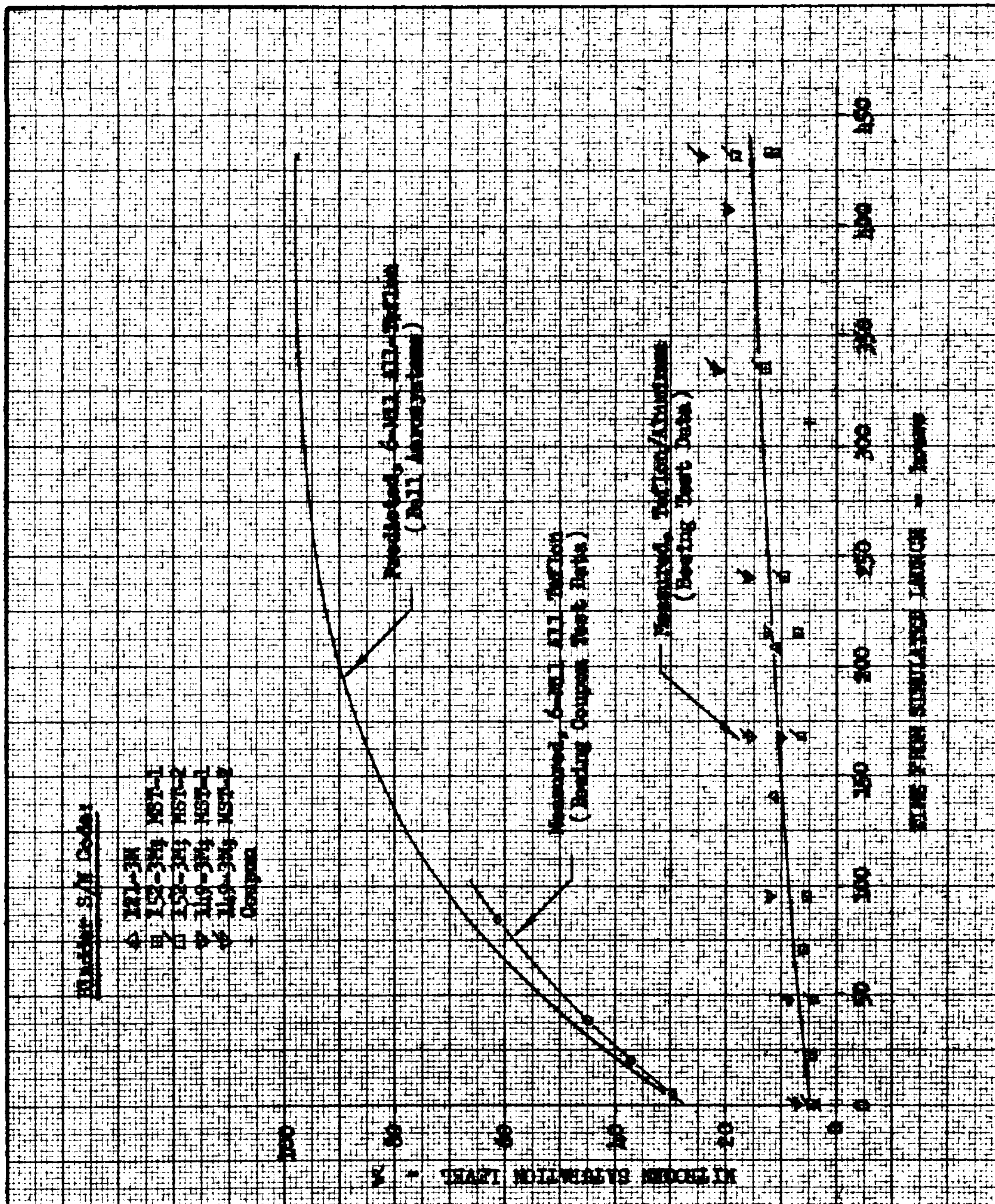
A major design goal of the composite teflon/aluminum expulsion bladder was that the aluminum interfoil impose a more impervious barrier to gas transmission and yet be thin enough to withstand repetitive cycling and vibration test without cracking or tearing. The rate at which nitrogen gas diffuses through the composite bladder should be low to the degree that the critical saturation level is not approached at the time of the final spacecraft maneuver. The maximum time increment is 32 days, 14 of which are pre-launch operations with the propellants pressurized to 45 psig.

The gas transmission characteristics of the teflon/aluminum bladder were evaluated by periodically withdrawing propellant samples from the test tankage at Tulalip and analyzing said samples to determine the amount of nitrogen in solution. Oxidizer samples were expelled, under pressure, into small containers (320-350 cc volume), delivered to Kent, and analyzed as described in Section 6.2.1. During the test program, 26 propellant samples were analyzed from three different tank assemblies. The nitrogen saturation level data, in percent, are plotted as a function of time at operating pressure in Figure 14. Comparable data for the standard all-teflon bladder (predicted and coupon measurements) are shown for comparative purposes. Observe that the saturation level is not zero at the start of the test. This "zero shift" results from two factors: 1) a degree of saturation, 3-4%, occurs in the process of transferring from the supply container to the propellant tank, and 2) the oxidizer becomes further saturated as a result of the 14-day pre-launch "soak" at 45 psig. Note that the saturation level at the time of launch is significantly greater for the all-teflon bladder. At the conclusion of the 32-day mission profile, the data indicate a nominal saturation level on the order of 18% (the final MST-2 values for Bladder S/N 149-3M are discounted in view of the unit's previous test history). The measured saturation levels are considerably less than that considered to be a critical value. The initial rate of nitrogen gas transmission (maximum partial pressure gradient) through a teflon/aluminum bladder has been found to be on the order of 0.006-0.01 scc/hr/in^2; in contrast, the transmission rate of a 6-mil all-teflon bladder is on the order of 0.2 scc/hr/in^2. These data adequately demonstrate the superior gas transmission Characteristics of the composite bladder construction.

6.2.3 Coupon Test Results

Gas transmission data have been obtained for all-teflon and teflon/aluminum bladder coupons. All-teflon sample tests were conducted to verify the saturation level growth curve as predicted by Bell Aerosystems. These data are plotted in Figure 14 and show satisfactory correlation with the predicted curve. The coupon test equipment exposed a bladder surface area of 8.612 sq. in. with a propellant reservoir of 307 cubic centimeters, a surface-volume ratio that closely approximates that of a complete bladder assembly, thereby eliminating the necessity of additional scaling factors.

Only one teflon/aluminum bladder coupon was exposed for test; the results are shown in Figure 14. Following the 17 January decision to incorporate the composite bladder in all flight spacecraft (Reference 8), further coupon testing was suspended in lieu of complete bladder test efforts.



	INITIALS	DATE	REV BY INITIALS	DATE	TITLE	MODEL
CALC	JC	3-11			NITROGEN SATURATION IN N_2O_4	Fig. 14
CHECK					EFFECTS OF BLADDER CONSTRUCTION	
APPD.						
APPD.						

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<u>Peel Strength Tests</u> - A standard teflon expulsion bladder is fabricated by spraying the teflon on a suitable mandrel; repeated spray passes are conducted until the desired thickness is achieved. The post-fabrication curing process tends to form the spray-pass lamina into a unitized structure. This does not hold for the composite teflon/aluminum bladder in that the presence of the aluminum foil introduces a foreign substance and there is a resulting reduction in bond strength between lamina. A series of coupon-level peel strength tests were conducted to evaluate the adhesive qualities of the teflon-aluminum bond.

Peel strength tests were conducted at the Kent Space Center. The procedure used was to subject the bladder coupon to nitrogen tetroxide for a period of 14 days, and then attempt to peel the teflon lamina away from the aluminum foil utilizing a Tinius-Olson Universal Tester in accordance with the general requirements of specification ASTM D-1876. Several exposed and unexposed (control) samples were tested in this manner: samples were peeled at a rate of ten inches per minute. The data thereby obtained are in terms of the force required to peel a one inch-wide sample at the aforementioned rate. The average peel strength of unexposed samples was found to be 1.96 lbs/inch; after 14 days exposure, the peel strength reduced to approximately 0.286 lbs/inch.

Though prolonged exposure to oxidizer reduced the teflon-aluminum adhesive strength characteristics, the test data indicated suitable retention of structural integrity and the absence of bladder delamination. Mission simulation testing, Section 6.3.2, revealed some localized delamination in Bladder S/N 149-3M which had been exposed to FAT and Qual vibration, six expulsion cycles, and 1629 hours of test in the presence of nitrogen tetroxide. The delamination may account for the larger saturation data shown for this unit in Figure 14; however, the results are fully acceptable and the measured leakage rate was only 0.5 scc/15 minutes.

6.3 <u>STORAGE & MISSION SIMULATION TEST EVALUATION</u>

Three tank/bladder assemblies successfully completed storage compatibility testing with nitrogen tetroxide. In the course of all storage testing, propellant samples were withdrawn for determination of nitrogen content, and expulsion cycles were conducted periodically. The following material summarizes this phase of the bladder test program.

6.3.1 <u>Tank S/N 9; Bladder S/N 121-3M</u>

The tank assembly was placed in test at the Tulalip Test Site at 1800 hours on 17 November. The unit was subjected to maximum operating conditions in the presence of nitrogen tetroxide for 208 hours (until 1000 hours on 26 November). Average test values of pressure and temperature were 231.5 psig and 82.4°F, respectively. A time history of the pressures and temperatures imposed on Tank S/N 9 are presented in Figure 15; the data are plotted at 6-hour time intervals.

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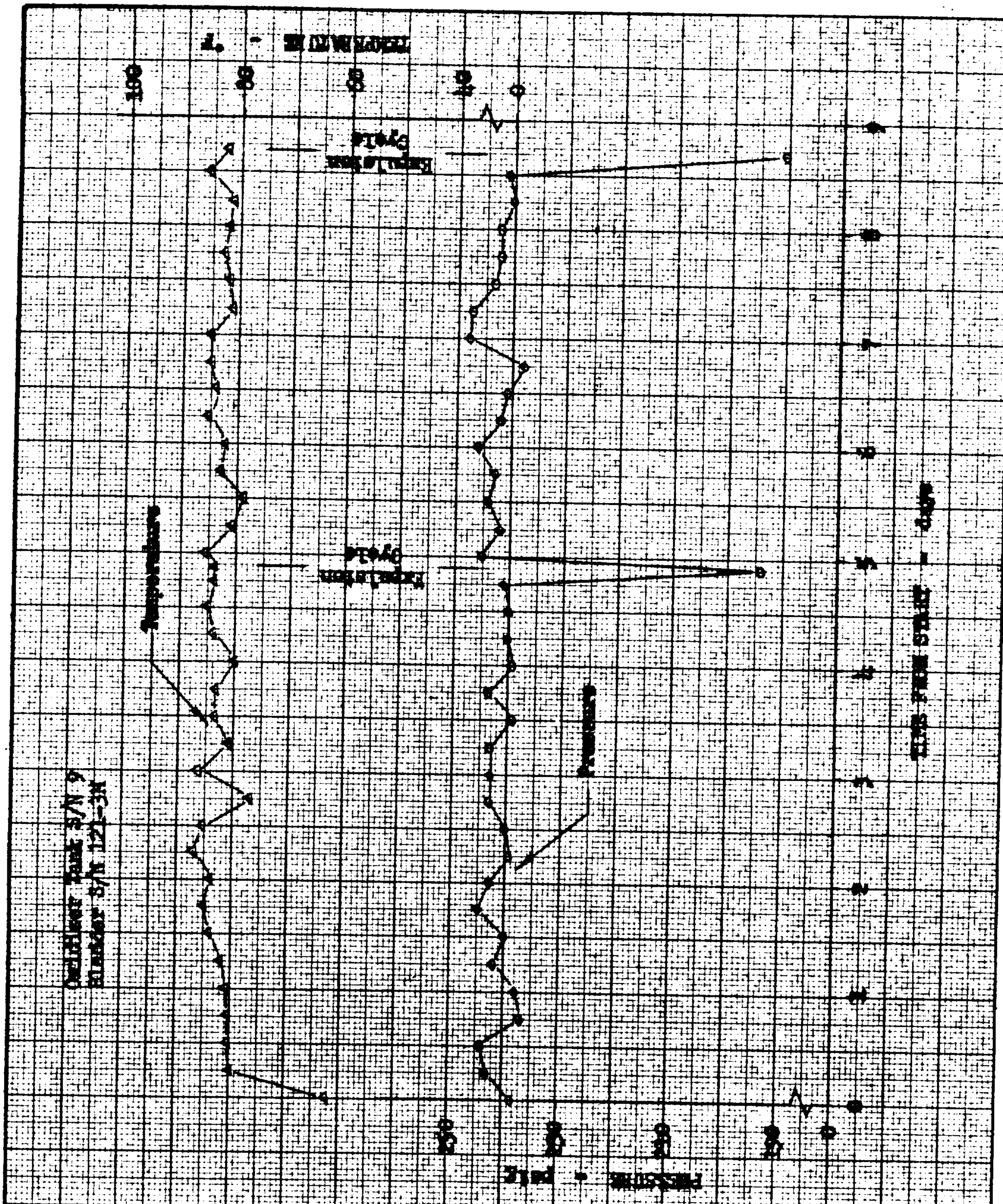

	INITIALS	DATE	REV BY INITIALS	DATE	TITLE	MODEL
CALC	JC	1-6-66			TEFLON/ALUMINUM EXPULSION BLADDER STORAGE TEST PRESSURE & TEMPERATURE HISTORY	Fig. 15
CHECK						
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The Reference 1 test plan specified the withdrawal of propellant samples and expulsion cycles on the fourth and eighth days of the test. A propellant sample was also withdrawn at the time of oxidizer loading to establish a "background" nitrogen content. Propellant saturation data are shown in Figure 14 (Section 6.2.2). A summary of the two expulsion cycle test conditions is presented in Table V.

Table V

BLADDER S/N 121-3M
EXPULSION TEST SUMMARY

Cycle Number	Pressure, psig	Temperature, °F	Flowrate, lbs/sec
1	193.9	84.2	0.117
2	190.3	82.4	0.085

Seven pounds of oxidizer were expelled in the first cycle, and the remaining oxidizer was expelled in the final cycle.

The assembly was flushed with Freon/methanol and returned to the MPC on 29 November; oxidizer had been present in the unit for a total of 281 hours. A bladder leakage test conducted upon receipt at the MPC resulted in a value of 1 scc/15 minutes. The results of the storage test on Bladder S/N 121-3M were not wholly satisfactory. Disassembly of the tank revealed several small areas where the aluminum foil had apparently crumbled and corroded. Figure 16 presents an overall photographic view of the bladder after removal and inflation; Figures 17 and 18 show close-up views of two major areas of aluminum deterioration. An unknown factor in the test results is that Tank S/N 9 had been nickel plated on the interior surfaces (a left-over unit from the tank storage program, Reference 2). A greenish-colored residue was visible in the tank interior and on the gas side of the bladder. X-ray diffraction analysis confirmed that the residue was a hydrated form of nickel nitrate, $Ni(NO_3)_2 \cdot 6H_2O$. Samples of the corroded aluminum foil were analyzed by infra-red spectrophotometer techniques and found to be a hydrated form of aluminum nitrate, $Al(NO_3)_3 \cdot 9H_2O$. The presence of the hydrated forms leads to the strong conclusion that the tank had somehow been contaminated with water vapor, possibly during a bladder leak test which involves a water displacement measurement method. The supplies of nitrogen and oxidizer were checked and found to be within specification in terms of water content. A product of the reaction between water and nitrogen tetroxide is nitric acid. Breakdown of the normal passive aluminum oxide layer and subsequent corrosion may have been enhanced by the presence of the nickel plating; nickel is known to be an active catalyst for many reactions. From the fact that other bladders passed mission simulation testing (Section 6.3.2) without deterioration, it is concluded that the abnormal condition of Bladder S/N 121-3M resulted from a procedural malfunction and an unnatural test environment; i.e., the presence of nickel.

Figure 16

BLADDER S/N 121-3M

ALUMINUM DETERIORATION,
FORWARD EXTREMITY

Figure 18

ALUMINUM DETERIORATION, AFT EXTREMITY

6.3.2 Mission Simulation Testing

Two tank/bladder assemblies were each subjected to two mission simulation tests in real time. The test units were Tank S/N 4/Bladder S/N 152-3M and Tank S/N 10/Bladder S/N 149-3M. The latter assembly had previously been subjected to FAT and Qual vibration, four expulsion cycles, and removal and re-installation of the bladder; hence, the results from this unit would represent an extreme case. From the typical spacecraft time-line analysis, the mission simulation profile presented in Table VI was implemented in this phase of the program.

Table VI

MISSION SIMULATION TEST PROFILE

Time, Days	
T - 14	Initiate pre-launch pad operations simulation. Condition test unit to 45 psig and 60°F.
T - 0	Simulate spacecraft launch. Increase tank pressure to 240 psig and simulate trans-lunar temperature environment of 80°F.
T + 1	Expulsion cycle simulating midcourse maneuver - 5 seconds duration.
T + 3	Expulsion cycle simulating midcourse maneuver - 50 seconds duration.
T + 4	Expulsion cycle simulating orbit injection maneuver - 615 seconds duration. Reduce temperature to 60°F to simulate nominal lunar orbit environment.
T + 18	Expulsion cycle simulating orbit transfer maneuver - expel to 98% level.

Propellant samples were to be withdrawn periodically throughout the above 32-day test for determination of nitrogen content. During expulsion cycles, the tank pressure was reduced to a nominal value of 190 psig to simulate actual operating conditions. At the conclusion of the test, a measurement of bladder leakage rate was conducted.

After successfully completing FAT-level vibration, Tank S/N 4 was delivered to Tulalip and the first mission simulation test initiated at 1420 hours on 2 January, 1966. The previous test history for Tank S/N 10 is discussed in Section 6.1.2; the excellent condition of Bladder S/N 149-3M led to the decision to also subject this combination to mission simulation testing for the purpose of increasing the level of design confidence. As Bladder S/N 152-3M (Tank S/N4) was the newer of the and more nearly representative of a flight component, adherence to the Table VI profile was emphasized for the unit. If equipment conflicts arose, then Tank S/N 10 mission events were to be advanced or delayed to avoid interfereence with Tank S/N 4. Tank S/N 10 was placed in test at 1145 hours on 4 January.

The first mission simulation test for both tanks concluded at 1000 hours on 4 February without incident. The second simulation test was initiated at 1100 hours on 7 February and concluded at 0930 hours on 11 March, also without incident. A summary of the environmental test conditions is given in Table VII.

Table VII

MISSION SIMULATION TEST ENVIRONMENT

	Pre-Launch	Trans-Lunar	Lunar Orbit
Tank S/N 4, Bladder S/N 152-3M			
MST-1			
Time, hrs.	355	96.5	336
Pressure, psig	49.0	243.8	244.0
Temperature, °F	59.6	80.4	61.0
MST-2			
Time, hrs.	334	95.5	336.6
Pressure, psig	51.1	240.6	243.3
Temperature, °F	62.6	85.1	63.3
Tank S/N 10, Bladder S/N 149-3M			
MST-1			
Time, hrs.	309.5	168	264.5
Pressure, psig	42.0	220.1	240.8
Temperature, °F	58.9	72.6	60.5
MST-2			
Time, hrs.	334	96.0	336.5
Pressure, psig	48.7	241.0	242.4
Temperature, °F	62.3	84.7	63.1

The pressures and temperatures quoted in the above table are averages based on data points taken at 6-hour intervals. Figures 19 and 19a show pressure and temperature time histories for Tank S/N 4; comparable data for Tank S/N 10 are given in Figures 20 and 20a. The pressure fluctuations noted in Figure 20 are the result of small facility-plumbing leakages that could not be corrected without interrupting the test: the magnitude of the pressure changes became noticeably less as oxidizer was expelled and the ullage volume increased. Corrective measures were successfully instituted prior to the second test. The time history plots also include indication as to when expulsion cycles were conducted and when propellant samples were withdrawn. Results of propellant sample analyses are discussed in Section 6.2.2. A summary of expulsion cycle data parameters is contained in Table VIII.

A bladder leak test was conducted at the conclusion of each simulation test in accordance with the procedures of Reference 1. At the conclusion of MST-1, the measured leakage rate was zero on both assemblies; at the conclusion of MST-2, the leakage rates were 0-0.5 scc/15 minutes on Tank S/N 4, and 0.5 scc/15 minutes on Tank S/N 10. The leakage allowed by Reference 5 is 4.0 scc/15 minutes.

Both tank assemblies were flushed with Freon/methanol and returned to the MPC on 11 March. During this phase of the test program, oxidizer had been present in Tanks S/N 4 and S/N 10 for a total of 1630 hours and 1584 hours, respectively. Post-test disassembly revealed that both bladders were generally in excellent condition; some minute cracking of the aluminum was evident as previously discussed in Section 5.0. There was no distinguishable deterioration or corrosion, nor any tearing of teflon or aluminum lamina. Bladder S/N 149-3M exhibited localized delamination between the aluminum and outer FEP laminas in the region of the unit's aft extremity (i.e., flange end). Photographic views of Bladders S/N 152-3M and 149-3M are shown in Figures 21 and 22, respectively.

6.4 VIBRATION TEST DATA

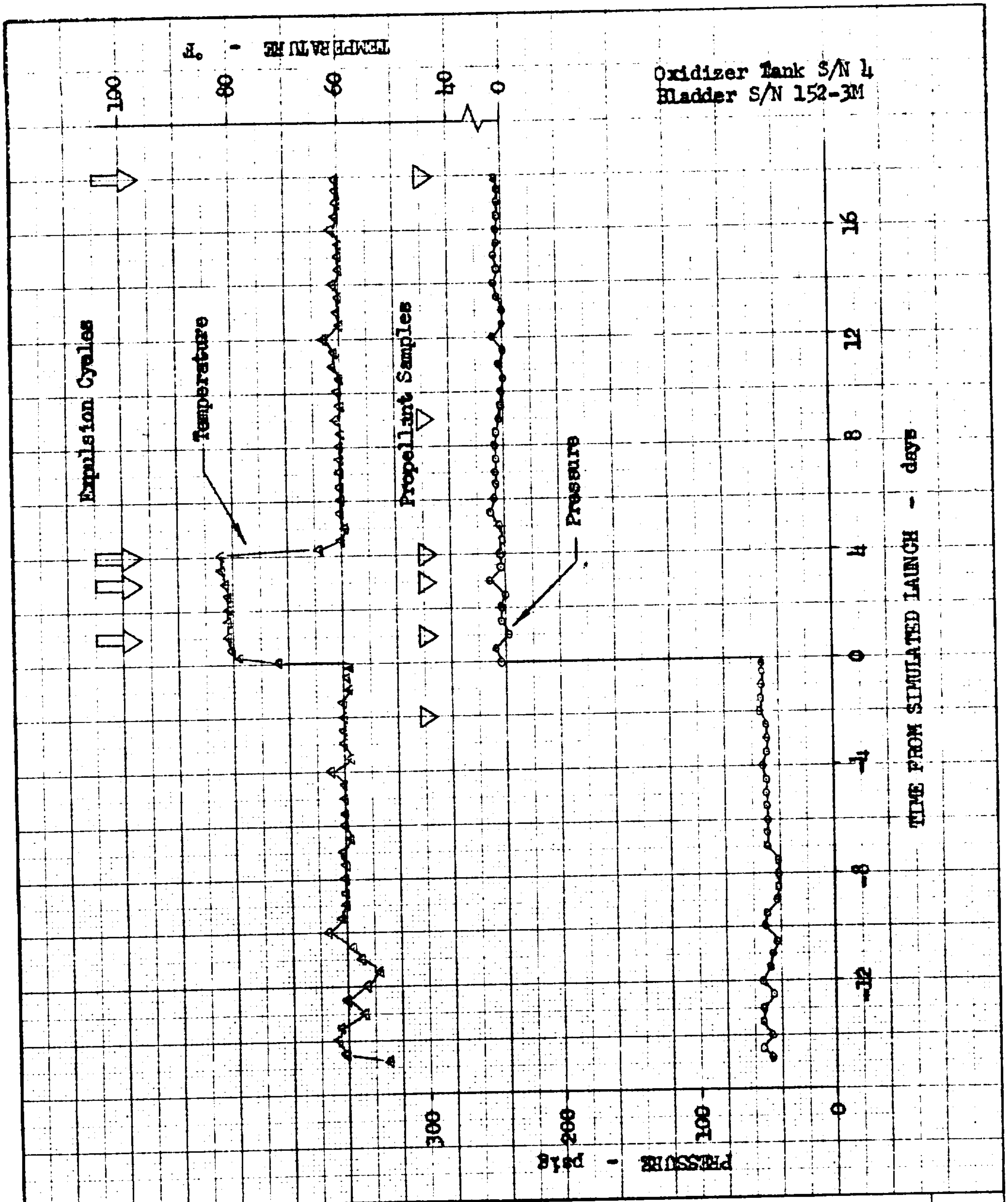
Four tank/bladder assemblies were subjected to vibration testing at the Kent Space Center. The environment to which the units have been tested are those specified in the oxidizer tank procurement specification, Reference 5. The facilities and procedures employed in vibration testing the teflon/aluminum expulsion bladder are discussed in Section 4.2 and Reference 1. Table IX summarizes the tank/bladder assemblies and the type of vibration spectrum to which each was tested.

Table IX

VIBRATION TEST SUMMARY

Tank S/N	Bladder S/N	Vibration Spectrum	Test Date
4	123-3M	FAT	11-29
10	124-3M	FAT	12-4
		Qual	12-4
10	149-3M	FAT	12-16
		Qual	12-17
4	152-3M	FAT	12-30

Note: FAT - Flight Acceptance Test
Qual - Qualification



	INITIALS	DATE	REV BY INITIALS	DATE	TITLE	MODEL
CALC	JC	2-7			TEFLON/ALUMINUM EXPULSION BLADDER MISSION SIMULATION TEST NO. 1 PRESSURE & TEMPERATURE HISTORY	Fig. 19
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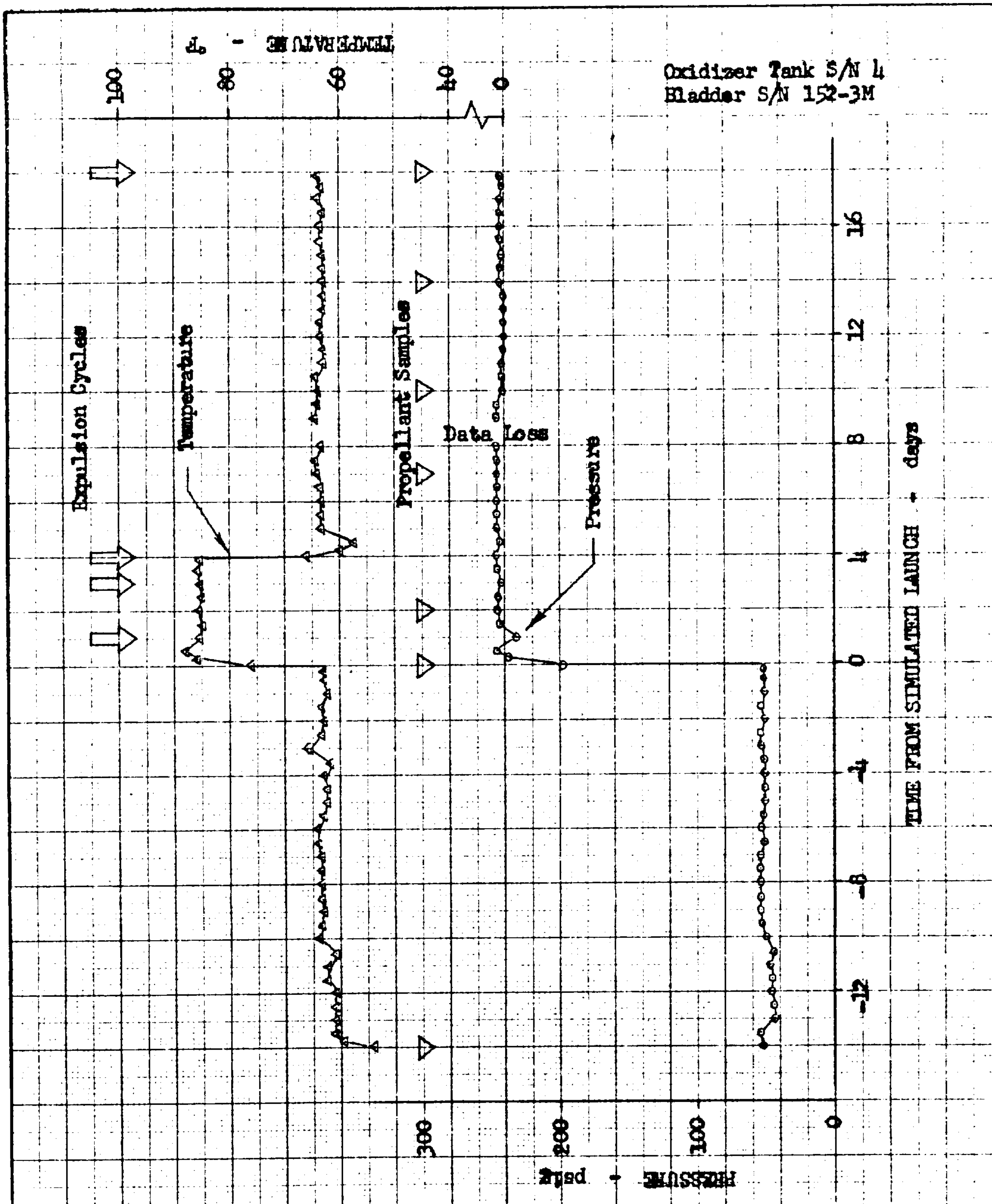

	INITIALS	DATE	REV BY INITIALS	DATE	TITLE	MODEL
CALC	JC	3-11			TEFLON/ALUMINUM EXPULSION BLADDER MISSION SIMULATION TEST NO. 2 PRESSURE & TEMPERATURE HISTORY	Fig. 19a
CHECK						
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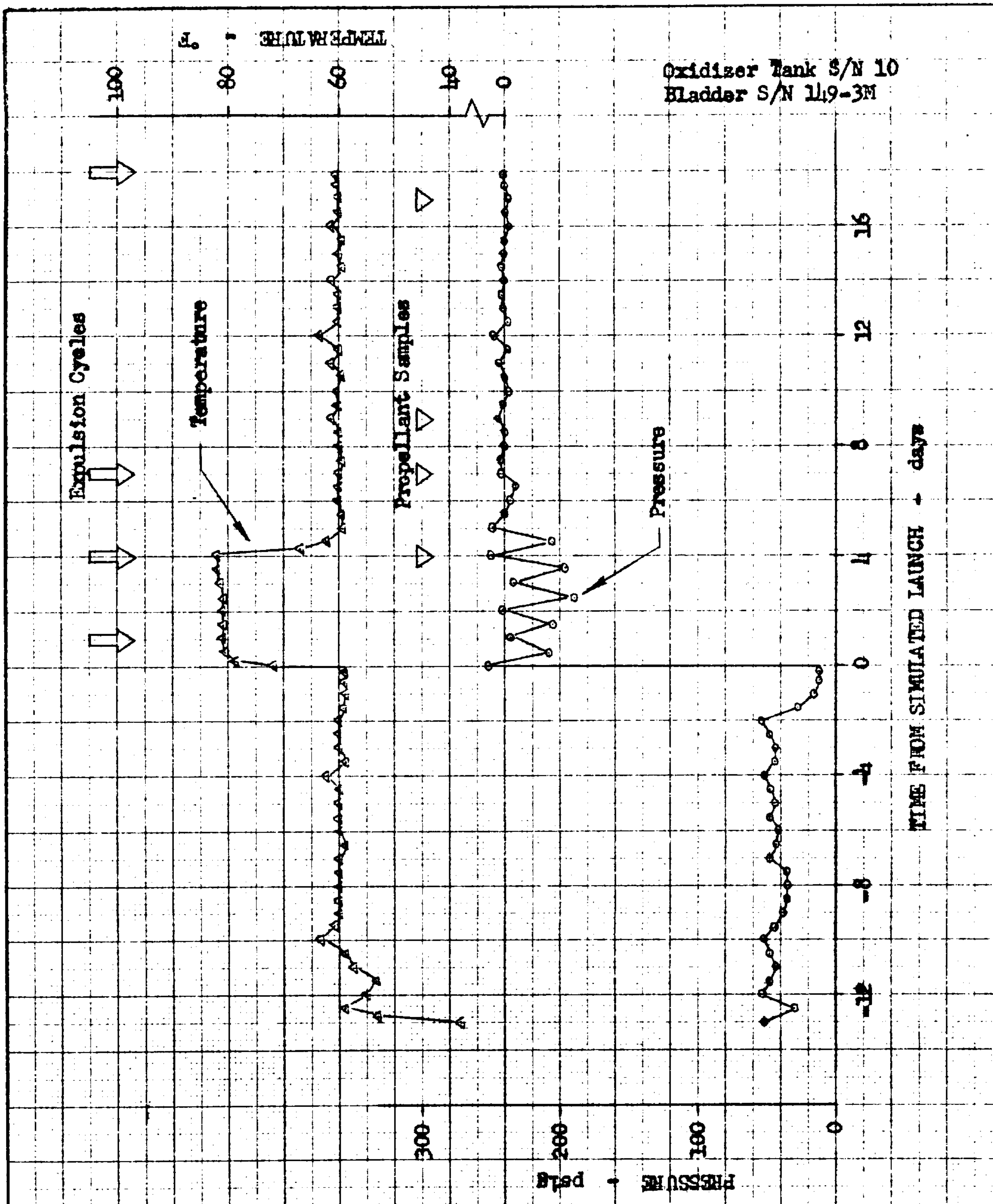

	INITIALS	DATE	REV BY INITIALS	DATE	TITLE	MODEL
CALC	JC	2-7			TEFLON/ALUMINUM EXPULSION BLADDER MISSION SIMULATION TEST NO. 1 PRESSURE & TEMPERATURE HISTORY	Fig. 20
CHECK						
APPD						
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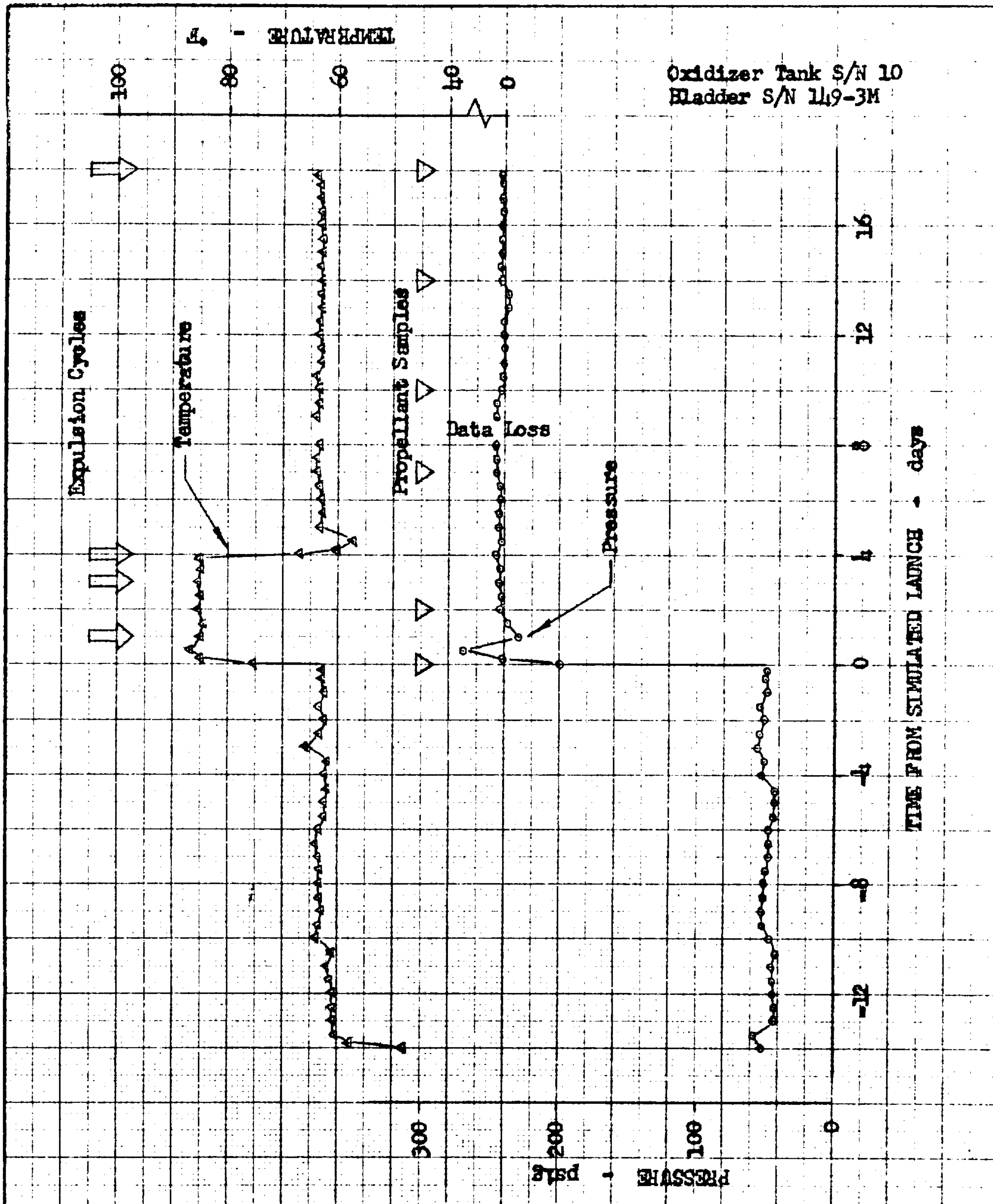

	INITIALS	DATE	REV BY INITIALS	DATE	TITLE	MODEL
CALC	JC	3-11			TEFLON/ALUMINUM EXPULSION BLADDER MISSION SIMULATION TEST NO. 2 PRESSURE & TEMPERATURE HISTORY	Fig. 20a
CHECK						
APPD						
APPD						

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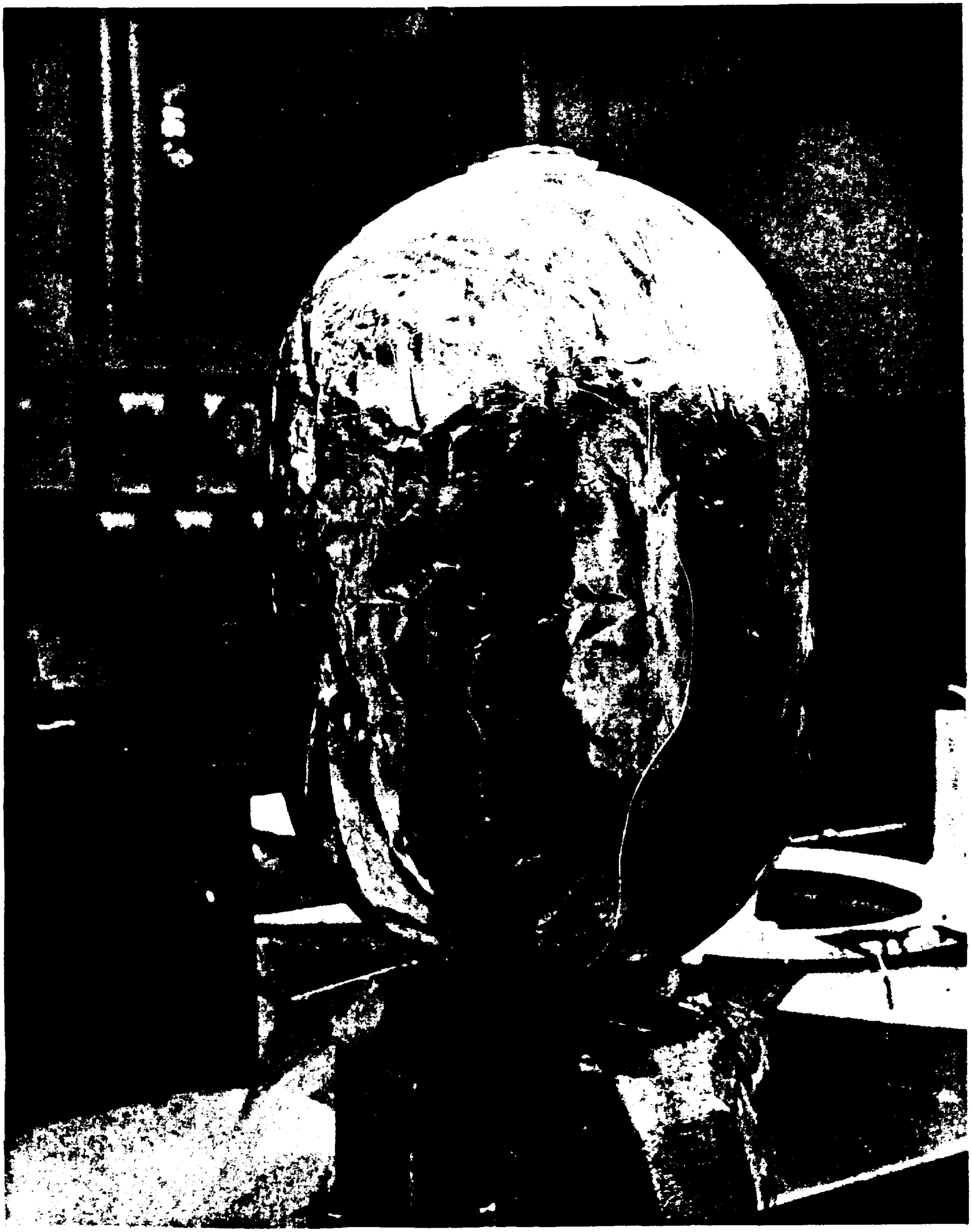
Table VIII

SIMULATED MANEUVER EXPULSION SUMMARY

	Pressure, psig	Temperature, °F	Flowrate, lbs/sec	Weight, lbs	Date
Tank S/N 4					
Bladder S/N 152-3M					
MST-1					
1st Midcourse	190	81	0.10	0.5	1-18
2nd Midcourse	207	80	0.045	2.25	1-20
Injection	201.2	82	0.074	61.0	1-21
Transfer	183.8	61	0.077	16.5	2-4
MST-2					
1st Midcourse	190	87.5	0.10	0.5	2-22
2nd Midcourse	191	84.5	0.14	7.0	2-24
Injection	188	85.4	0.143	70.0	2-25
Transfer	190.5	63.5	0.208	2.5	3-11
Tank S/N 10					
Bladder S/N 149-3M					
MST-1					
1st Midcourse	192	81	0.10	0.5	1-18
2nd Midcourse	214.5	82	0.14	7.0	1-21
Injection	185.7	60	0.108	66.5	1-24
Transfer	189.3	59.7	0.09	10.5	2-4
MST-2					
1st Midcourse	192	87.5	0.10	0.5	2-22
2nd Midcourse	193	84.5	0.13	6.5	2-24
Injection	190.2	85.1	0.114	70.0	2-25
Transfer	195.5	63	0.148	5.5	3-11

Figure 21



BLADDER S N 152-3M, POST TEST

Figure 22

BLADDER S N 149-3M. POST TEST

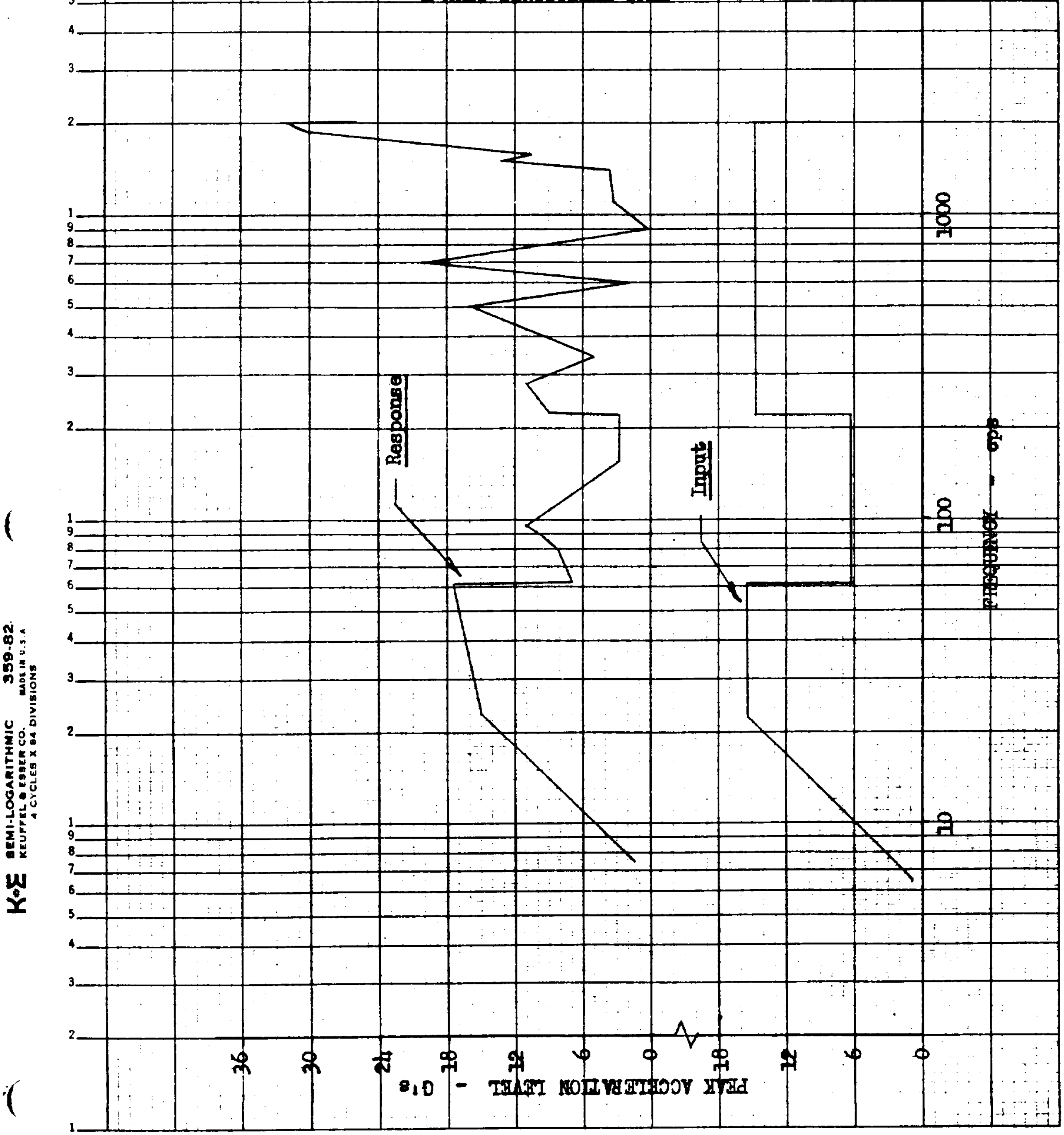
In both FAT and Qual level vibration testing, the test unit was subjected to appropriate sinusoidal and random vibration spectrums in each of the three principal axes. Figure 23 presents typical Qual-level sinusoidal input and tank response characteristics. Figure 24 shows a random input spectrum, and Figure 25 presents the manner in which the tank responded to that input. The recorded tank response data agree favorably with that obtained by Bell Aerosystems.

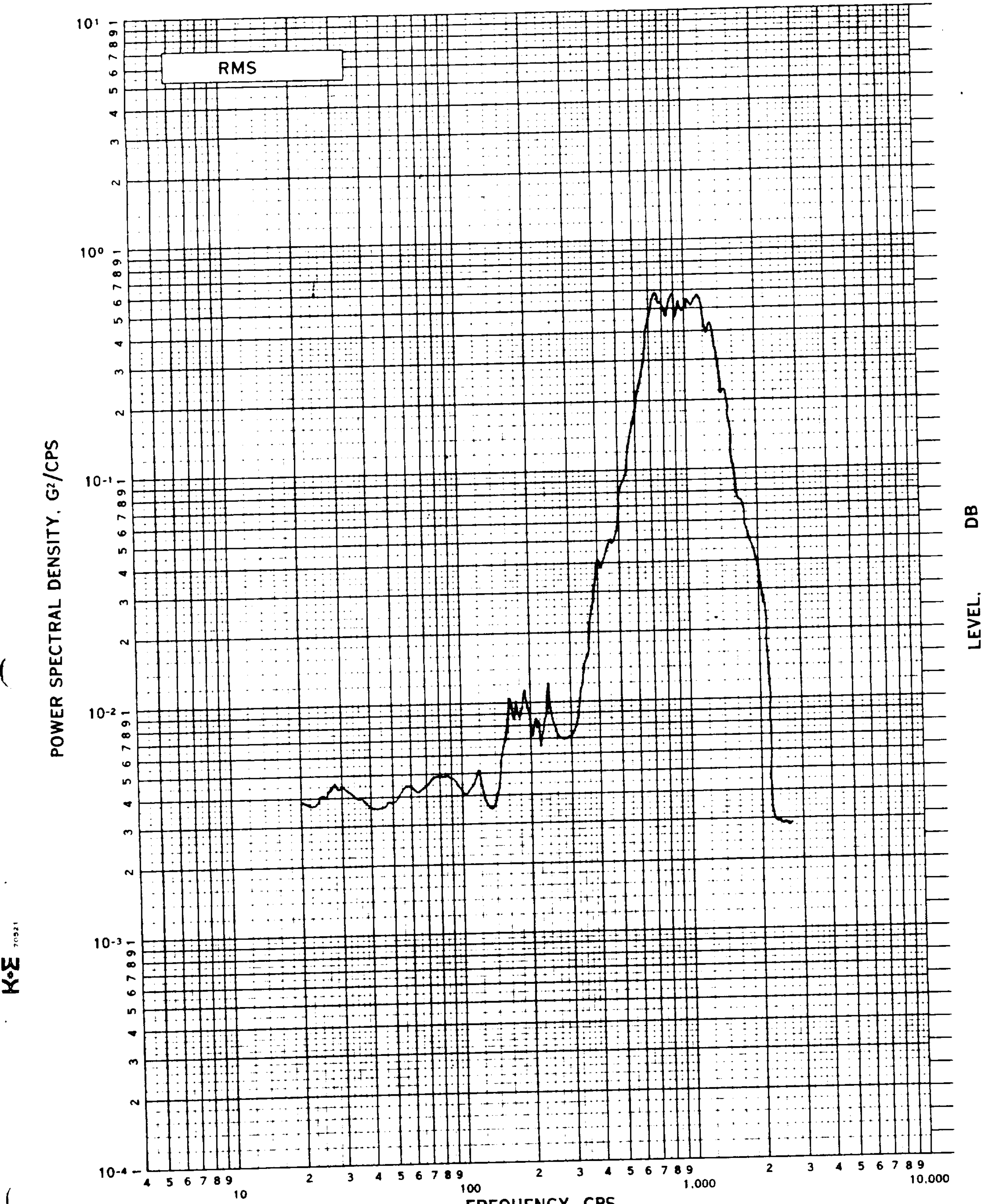
One bladder failure was observed upon completion of vibration testing; however, it cannot be established if the failure is attributable to vibration test, or improper installation methods. After completing Qual-level testing, the leakage rate across Bladder S/N 124-3M was found to be 10 scc/15 minutes; this is 2-1/2 times the specification allowable. Zero leakage had been measured prior to vibration test. Disassembly and inspection did not indicate any damage to the unit, but it was noted that the bladder was permanently twisted, with respect to the propellant standpipe, at both the forward and aft attach points. The bladder assembly was leak tested at an internal pressure equivalent to ten inches of water - no significant leakage could be detected at any location with a helium leak detector. The tank/bladder combination was reassembled and again leak tested; the leak rate had increased to 20 scc/15 minutes. As the leakage could not be eliminated, the unit was rejected and Bladder S/N 149-3M was installed into Tank S/N 10.

On all other bladder assemblies, the measured leak rate following vibration test was found to be zero. It is suspected that the failure of Bladder S/N 124-3M was attributable to a slightly improper installation that was aggravated by vibration test.

Figure 23

TEFLON/ALUMINUM BLADDER VIBRATION
TANK S/N 10; BLADDER S/N 149-3M
X-AXIS SINUSOIDAL QUAL





DATE

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CHECK
APP'D
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TEFLON/ALUMINUM BLADDER VIBRATION
TANK S/N 10; BLADDER S/N 149-3M
X-AXIS RANDOM QUAL INPUT

BOEING
SEATTLE

Fig. 24
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PAGE: 49

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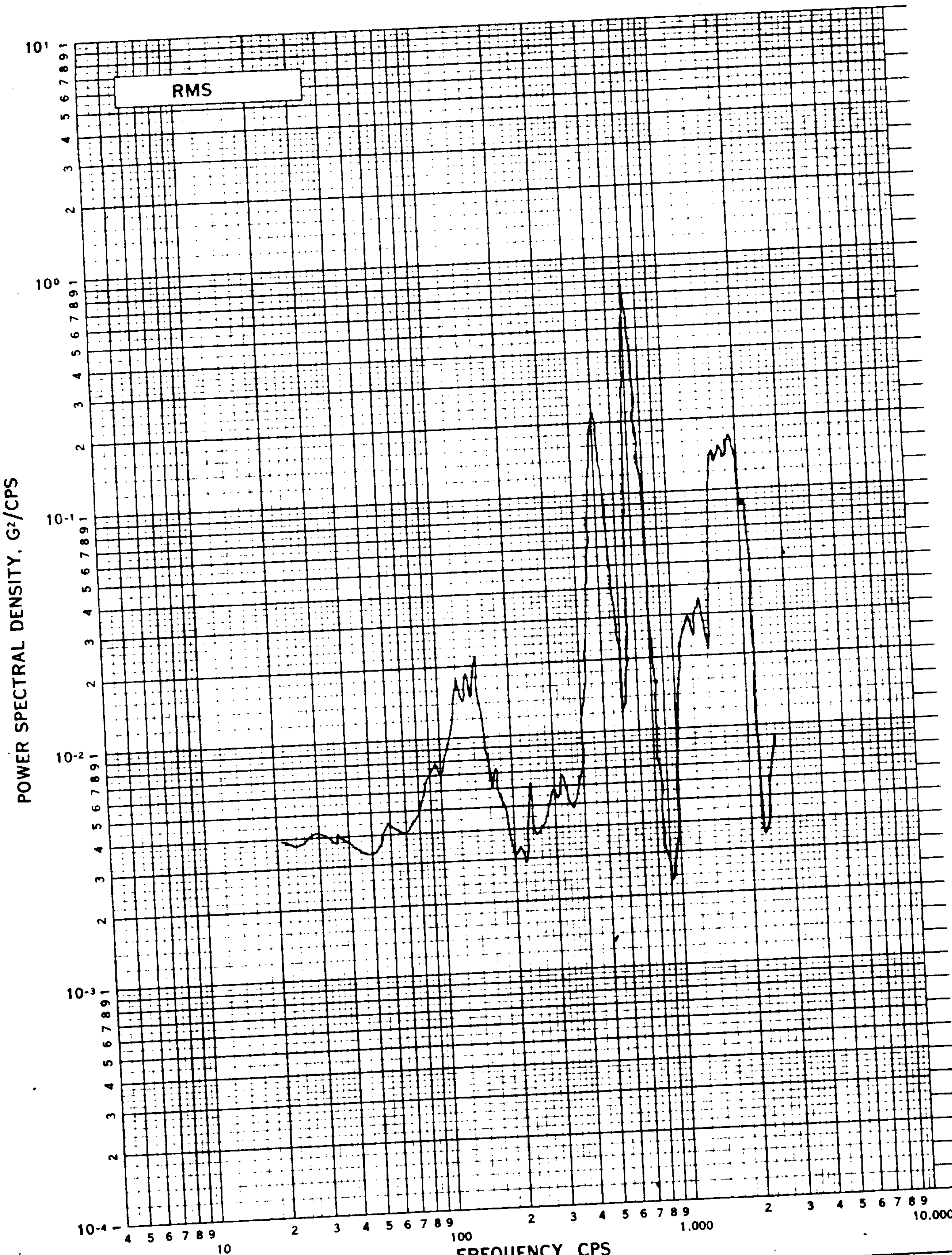

DATE

CALC.	TEFLON/ALUMINUM BLADDER VIBRATION
CHECK	TANK S/N 10; BLADDER S/N 149-3M
APP'D	X-AXIS RANDOM QUAL RESPONSE
APP'D	BOEING
APP'D	SEATTLE

Fig. 25

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7.0 CONCLUSION

The desirable characteristics of a positive expulsion system are that there by a minimum rate of permeation and gas transmission, light weight, and repetitive cyclic capability. A plastic or rubber expulsion bladder has the advantage of the latter two characteristics but is deficient in the first, a metallic bellows design imposes a weight penalty, and a metallic bladder is not capable of repetitive cycling. The design goal of the composite teflon/aluminum expulsion bladder was to attain the most favorable compromise of the desirable characteristics. The test results as reported herein indicate that this goal has been achieved. A cumulative summary of the four test units is presented in Table X.

Table X

CUMULATIVE TEST SUMMARY
FOUR BLADDERS

Number of 90% Expulsions	4
Number of 98% Expulsions	9
Total Exposure to N_2O_4	3658 hours.
3-Axis FAT Vibration	3 units
3-Axis Qual Vibration.	1 unit
Approximate Vibration Time	55 minutes
Number of Permeation Samples	26

Bladder S/N 149-3M accumulated the largest, and most varied, amount of test activity including 1) FAT and Qual vibration (30 minutes), 2) two 90% and four 98% expulsion cycles, and 3) 1629 hours total exposure to nitrogen tetroxide. The post-test condition of the unit was satisfactory. The teflon/aluminum bladder concept achieved a major design goal of reducing the rate of nitrogen gas transmission; the nominal saturation level after 32 days of mission profile testing is on the order of 18% as compared to 100% with an all-teflon bladder in the same time period.

On the basis of the reported test data, The Boeing Company recommended, and NASA-Langley concurred, that teflon/aluminum bladders be incorporated in the oxidizer tanks of all flight spacecraft.